

The Journal of the **INSTITUTE OF METALS**

and
METALLURGICAL ABSTRACTS



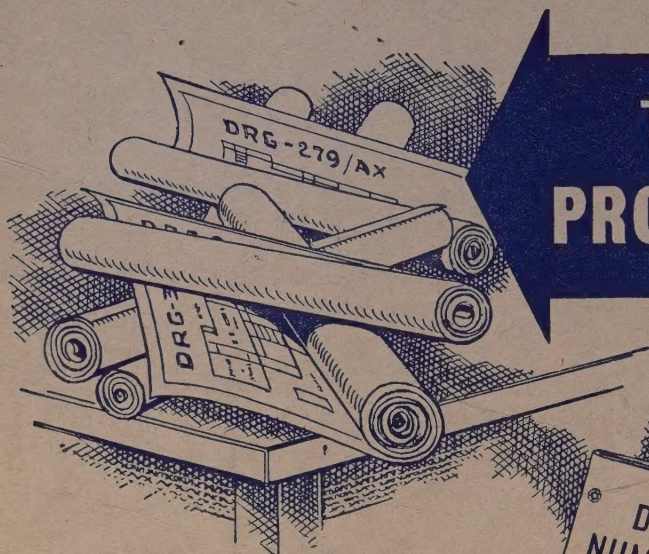
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| 1138. The Solubility of Hydrogen in Liquid and Solid Aluminium. C. E. Ransley and H. Neufeld | 599 |

DISCUSSION AND CORRESPONDENCE
ON PAPERS PRESENTED TO THE
INSTITUTE 621

Papers Nos. 1108 (Lacombe and Beaujard);
1111 (Cook and Larke); 1112 (Northcott,
McLean, and Lee); 1113 (McLean); 1117
(Brenner and Roth); 1119 and 1120 (Jennings,
Singer, and Pumphrey; Jennings and
Pumphrey); 1121 (Latin); and 1122 (Dud-
zinski, Murray, Mott, and Chalmers).

AUGUST 1948

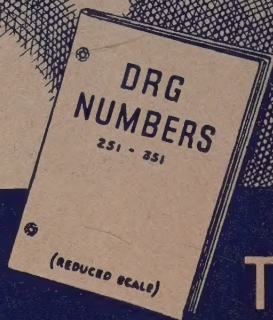


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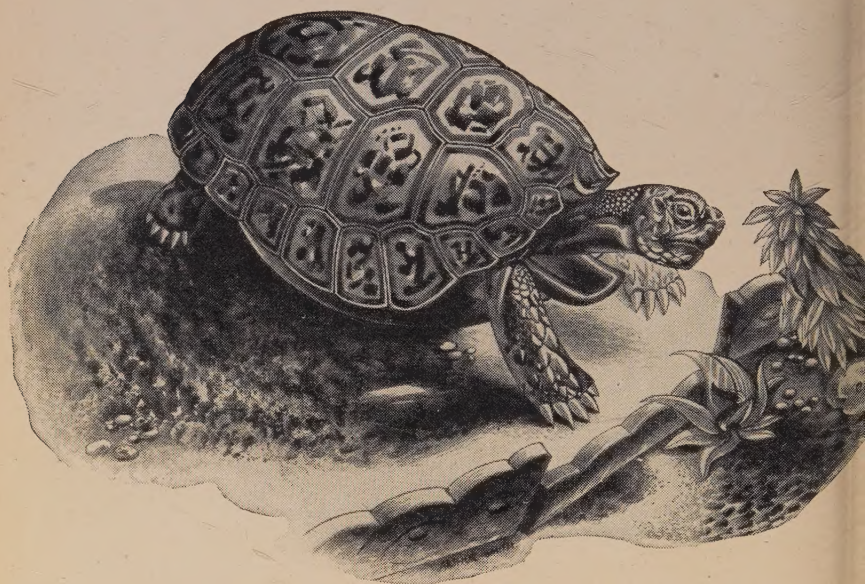
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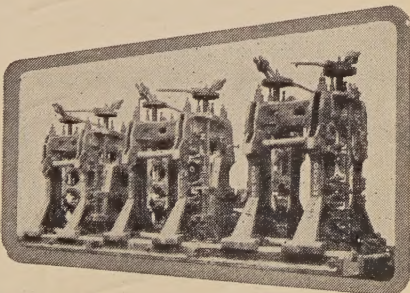
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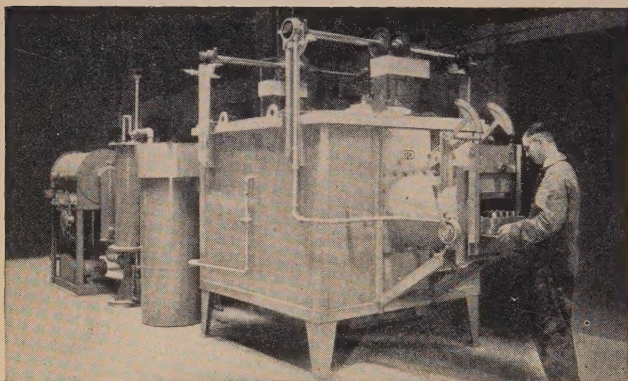
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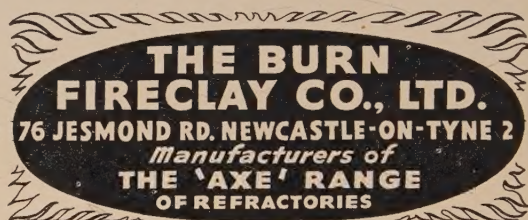
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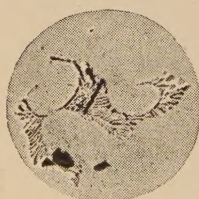
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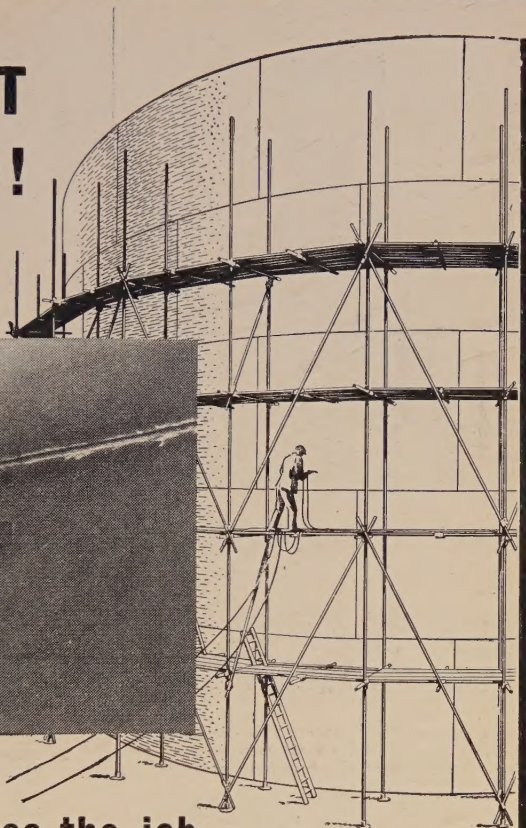
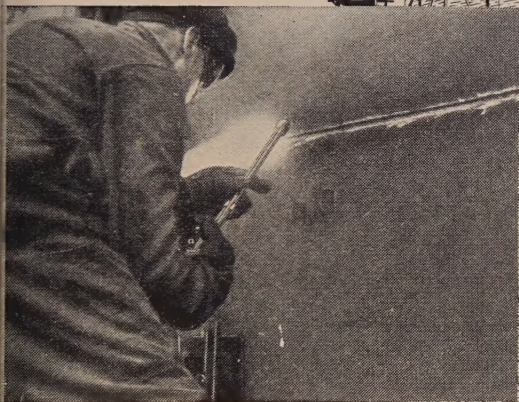


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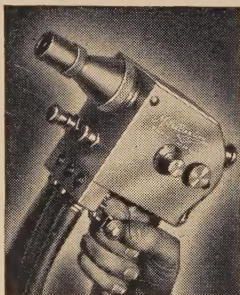
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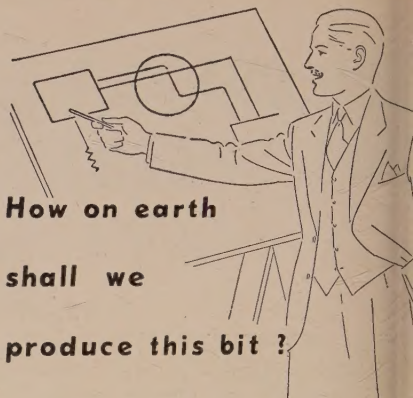
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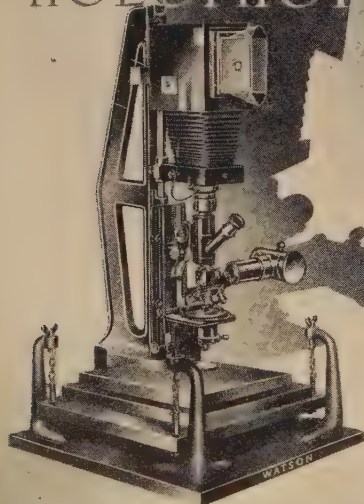
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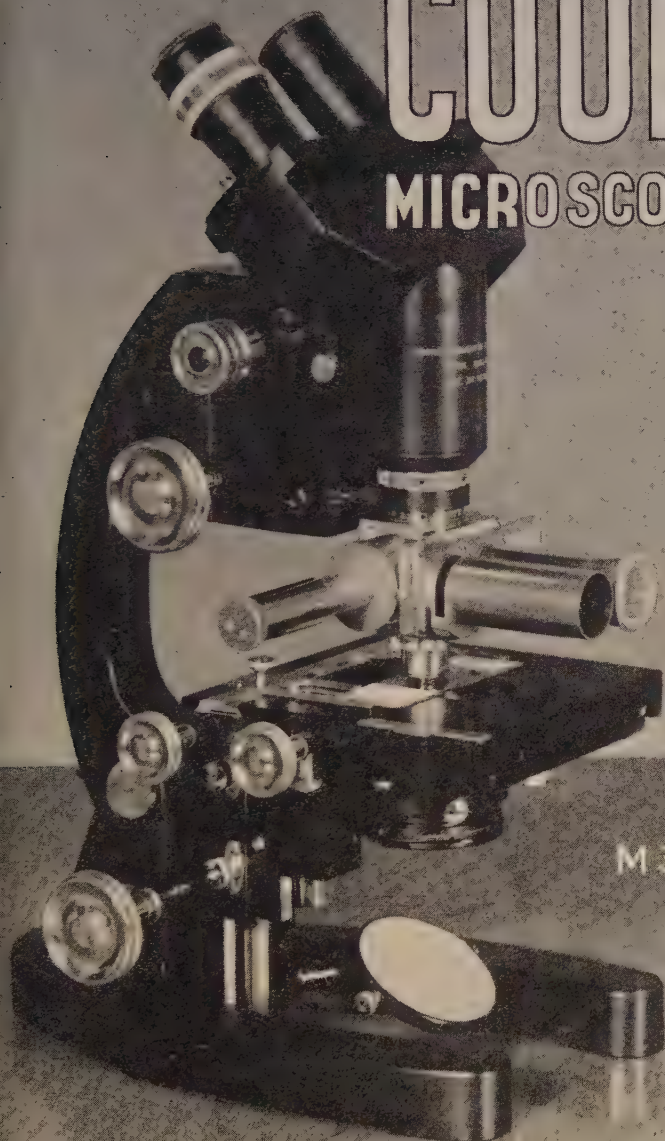
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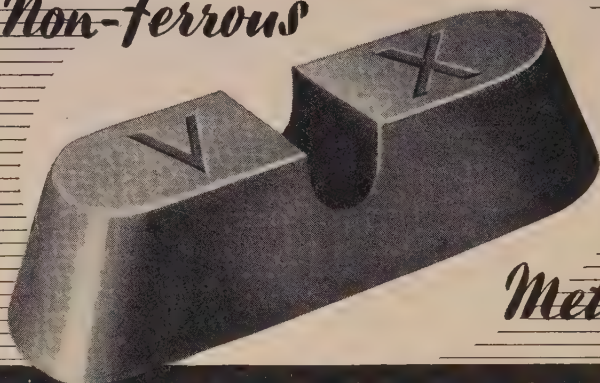


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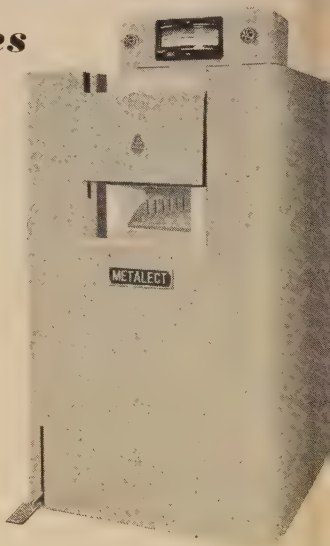
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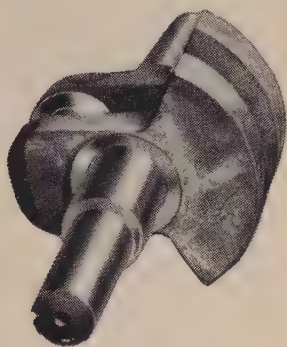
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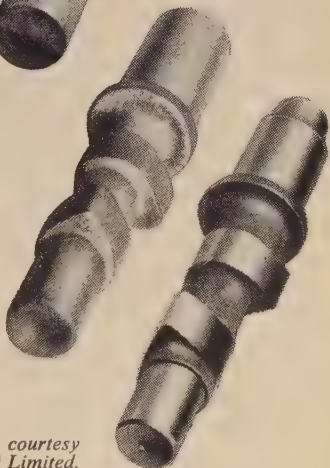


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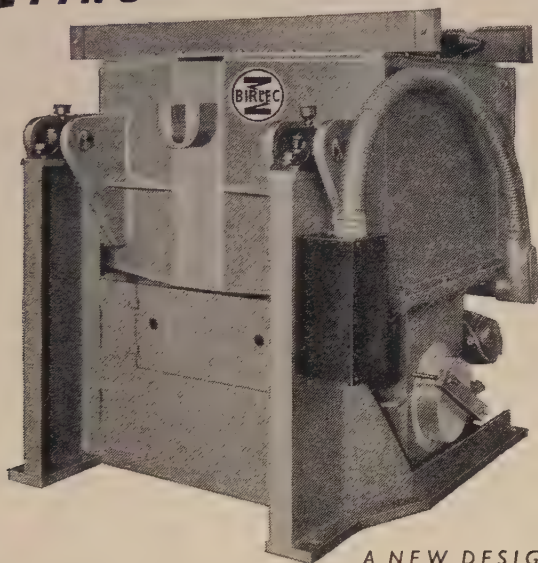
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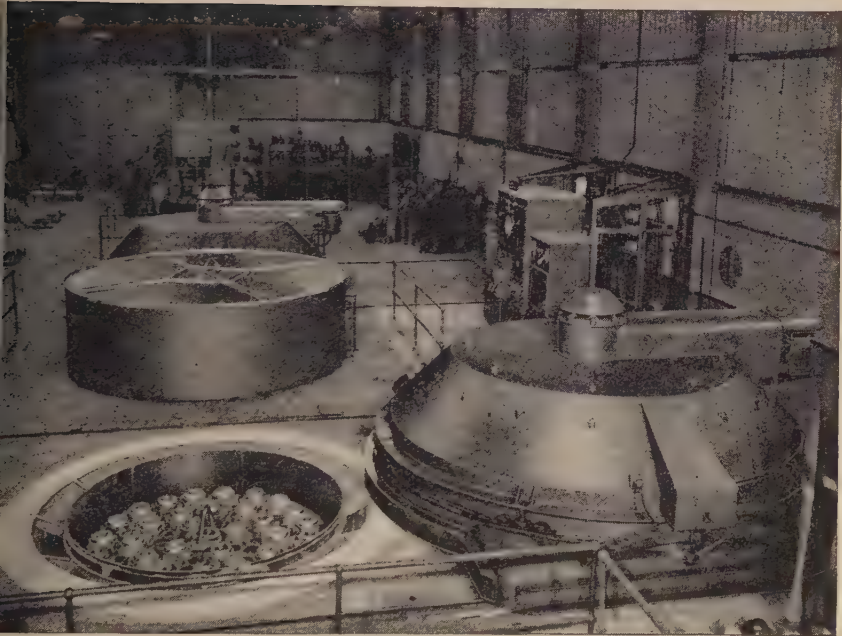
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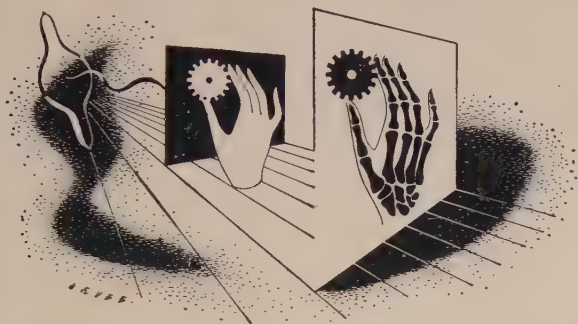
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THE SOLUBILITY OF HYDROGEN IN LIQUID AND SOLID ALUMINIUM.*

1138

By C. E. RANSLEY,† M.Sc., Ph.D., MEMBER, and H. NEUFELD,†
STUDENT MEMBER.

SYNOPSIS.

The solubility of hydrogen in liquid aluminium of high purity has been re-determined by a standard method. The results obtained indicate that over the temperature range 670°–850° C. the solubility S (c.c./100 g. metal) is given by the equation :

$$\log_{10} S/p^{1/2} = -2760/T + 1.356$$

where p is the hydrogen pressure in mm. of mercury and T is the absolute temperature. At temperatures approaching the freezing point, the new values are considerably higher than those previously accepted, and it is pointed out that this is more concordant with practical observations.

The solubility in the solid metal has not previously been measured accurately. It is very small, but has been determined with reasonable accuracy by a saturation and quenching technique. The square-root law relating solubility to the hydrogen pressure has been confirmed and the general results over the range 465°–620° C. can be represented by the equation :

$$\log_{10} S/p^{1/2} = -2080/T - 0.652$$

It is shown that the porosity of a standard sand-bar cast in commercial-purity aluminium is a linear function of the hydrogen content of the metal, and the experimental observations are discussed from the point of view of the new solubility data.

I.—INTRODUCTION.

THE solubility of hydrogen in liquid aluminium has been measured by Czochralski,¹ Iwasé,² Röntgen and Braun,³ Röntgen and Möller,⁴ Bircumshaw,⁵ Baukloh and Oesterlen,⁶ Winterhager,⁷ and Baukloh and Redjali.⁸ The results of the first two authors can probably be disregarded. The data of the remainder tend to converge at higher temperatures (1000°–1200° C.), but show considerable divergence at temperatures near the freezing point of the metal. This is illustrated by the following table of actual determinations at or about 700° C.

Authors.	Temperature, ° C.	Solubility (c.c./100 g.) at 1 atm. pressure.
Röntgen and Braun . . .	700°	0.08 *
Röntgen and Möller . . .	690°	0.27
Bircumshaw	700°	0.23
Baukloh and Oesterlen . . .	700°	0.95
Baukloh and Redjali . . .	700°	0.45

* Manuscript received 16 April 1948.

† Research Laboratories of the British Aluminium Co., Ltd., Gerrards Cross, Bucks.

The generally accepted value at 700° C. is about 0.25 c.c.,^{9*} and the interpolated value at 730° C., a normal holding temperature for pure aluminium and its alloys during melting, is about 0.4 c.c.

There appeared to the writers to be a considerable amount of indirect evidence to suggest that this value is too low, and since it is impossible to develop any precise picture of the effects of hydrogen in aluminium without a sure background with respect to solubility data, it was decided to re-determine the values in the important temperature region (660°–850° C.).

No systematic measurements of the solubility in solid aluminium have previously been made. It is too small to be detected by experiments with the type of apparatus used for the liquid metals, and is usually reported as nil.³ Eborall and Ransley¹⁰ estimated the solubility in commercial aluminium at 600° C. to be approximately 0.10 c.c./100 g., but their method involved the subtraction of a large and rather uncertain correction from their measured value. An indirect assessment of the solubility at the freezing point should be possible from the extrapolation to zero porosity of a porosity–hydrogen content relationship for cast bars. Metcalfe,¹¹ by this type of procedure, arrived at a figure of 0.09 c.c. It is not possible to say, however, to what extent supersaturation of the solid occurs under these circumstances. The writers have recently carried out a number of porosity and hydrogen-content measurements on standard sand-cast bars, and the results of these tests will be discussed in a later section of this paper.

The standard method for the determination of the solubility of gases in molten or solid metals is to make a direct measurement of the volume of gas absorbed by a known weight of metal contained in a bulb of calibrated volume. The “dead space” of the vessel over the range of temperature to be covered is usually calibrated by means of an inert gas, such as argon, which is not dissolved by the metal. The desired quantity is thus derived from the difference of two relatively large volumes, and when the solubility is small, as it is in liquid aluminium at low temperatures, the method leaves much to be desired. Accurate measurement can only be obtained by reducing the dead space to as small a volume as possible. It is also essential that solution of the gas in the crucible containing the metal, and in the material of which the bulb is constructed, should be either negligible or at least known with sufficient accuracy to enable suitable correction to be made.

In spite of these difficulties, the direct-absorption method appears

* Unless otherwise stated, the solubility and gas-content data given in this paper are expressed as c.c. (at 20° C. and 1 atm. pressure) per 100 g. of metal. The solubility results normally refer to the value in equilibrium with 1 atm. pressure of hydrogen.

to be the only positive one available for measurements on liquid metals; possible alternative methods have been examined without success. One which was tried, involving the sudden exhaustion of the atmosphere above the metal, followed by degassing of the metal itself, failed because of the instability of the melt, which manifested itself by an immediate and sometimes disastrous bubble formation. Quenching of the liquid metal after it has been saturated by contact with the desired atmosphere, followed by a determination of the gas retained in solid solution, is a more feasible method, but is only admissible when it can be demonstrated by independent experiment that there is no loss of gas by diffusion during the quench.

The direct-absorption method is obviously not suitable for measurements of the solid solubility, because the volume of hydrogen dissolved is undoubtedly very small. In this case, however, saturation, quenching, and subsequent extraction of the dissolved gas can be used with more confidence, since the loss of gas by diffusion can be readily assessed. In developing a method on these lines, however, it was considered essential to design the apparatus so that a number of determinations could be carried out on one specimen without the necessity of any intermediate exposure to the atmosphere.

II.—THE SOLUBILITY OF HYDROGEN IN LIQUID ALUMINIUM.

1. *Apparatus.*

There were only two materials available which were suitable for use for the construction of the absorption vessel:

(a) *Boro-Silicate Glass.*—This is a special hard glass supplied by the General Electric Co., Ltd. A preliminary test showed that the solution of hydrogen in this glass and the rate of permeation through it are sufficiently small to be negligible. Unfortunately, it will only withstand 690° C. under vacuum conditions, and this severely limits the range of temperature over which it can be used for determinations on molten metal.

(b) *Clear Silica Glass.*—This has frequently been used for this type of apparatus, e.g. by Bircumshaw. Hydrogen diffuses quite rapidly through silica at temperatures above 600° C., and it is customary to surround the absorption vessel with an outer jacket, which is filled with hydrogen at the same pressure as that in the inner bulb, and thus compensate for any outward flow of hydrogen. It should be noted, however, that this device does not prevent some solution of hydrogen in the glass from the inside. Since the solubility of hydrogen in silica is quite

high (0.01 c.c. N.T.P./c.c. of silica, not varying greatly with temperature), it can amount to an appreciable fraction of the observed solubility in the sample. In spite of its importance, this correction has been omitted by most workers.

After some preliminary experiments, a suitable form of apparatus was evolved, which is shown in Fig. 1. The construction of the

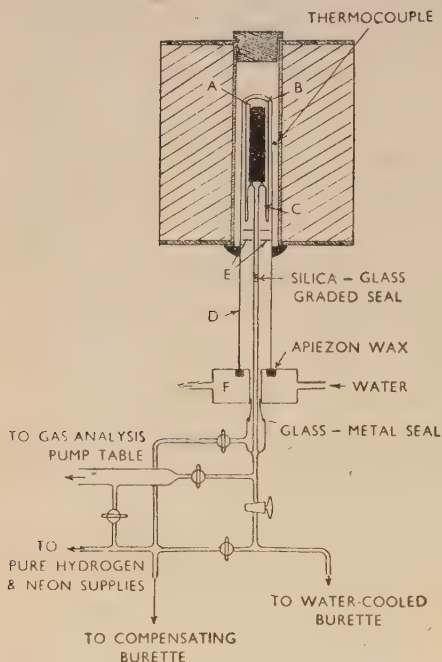


FIG. 1.—Apparatus for Measurements on Liquid Metal.

via an internal seal on a glassed copper thimble soldered into the bottom of *F*. The outer bulb *D*, which fitted over the internal assembly with a clearance of 1–2 mm., was sealed with Apiezon wax into a groove in the top of the metal head.

This bulb was heated by a silica-tube furnace wound to compensate for end losses and thus give a sufficiently uniform high-temperature zone. Two internal nickel discs *E* served as radiation screens where the bulb emerged at the bottom of the furnace; the top of the furnace was closed by an insulating plug. The temperature of the sample was measured by a Chromel–Alumel thermocouple wired on to the bulb *D*

the absorption vessel shown in the sketch made it possible to seal the metal charge into a bulb without overheating and at the same time enabled the dead space to be kept down to a minimum. The metal was in the form of a freshly turned cylinder weighing 30–40 g., which was placed in a crucible *A*, 20 mm. dia., 85 mm. long, made of recrystallized alumina (ΔRR , supplied by the Morgan Crucible Co.). This was introduced into the cylindrical bulb *B*, and the umbrella-shaped base *C* was then inserted and sealed round the skirt with a small flame.

The absorption vessel was supported by the central small-bore tube as shown; this passed out through the water-cooled metal head *F*,

at the mid-point of the crucible. This couple was checked in early experiments against another one inserted through the head *F* and tied on to the inner bulb. The operation of the furnace was controlled by an energy regulator which enabled the temperature to be kept constant to within $\pm 5^\circ \text{C}$.

The arrangement of the burettes calls for little comment. The one for admission of gas to the metal consisted of a 25-c.c. burette which could be read to ± 0.01 c.c., and was water-cooled to maintain it at a constant temperature. The volume of the connection from the burette to the absorption bulb was kept as small as possible, and in most experiments the total dead space was of the order of 10 c.c. The mercury level in this burette, and in the comparison burette required for the regulation of pressure, was controlled smoothly by adjusting the height of a mercury reservoir. The comparison burette was connected at the top to a large air reservoir and a mercury monometer, so that the pressure in the burette could be adjusted to any desired value; actually all the final measurements were carried out at a pressure of 760 mm. of mercury. The outer jacket *D* was connected to a simple U-tube burette with an adjustable mercury reservoir so that the pressure in this bulb could be adjusted to be equal to that in the absorption vessel.

As indicated in Fig. 1, the whole apparatus was connected to a gas-analysis pump table of a type previously described,¹⁰ i.e. a two-stage diffusion pump discharging into a low-pressure analytical system backed by a three-stage diffusion pump. This arrangement was very convenient, since not only could the apparatus be pumped to a pressure of 10^{-6} mm., but it was also possible, when required, to collect and measure the gas evolved from either the sample vessel or the outer jacket.

2. Materials.

The metal used in all the experiments was taken from a selected batch of super-purity aluminium, which had been analysed as follows :

Si, %.	Fe, %.	Cu, %.
0.0005	0.0005	0.0005

Prior to use it was melted in a graphite crucible and maintained at about 850°C . in a high vacuum for 3–4 hr. This was done to ensure that the metal was sufficiently free from any elements which might tend to volatilize during the solution experiments and thus possibly “getter” a certain amount of hydrogen.

Neon was chosen as the inert gas for dead-space calibration, in preference to the more commonly used argon, because (*a*) it could be

purified on the pump table very simply by passage through an activated charcoal tube cooled in liquid air, and (b) since its thermal conductivity is higher than that of argon, it is more nearly equivalent to hydrogen in its behaviour. The neon used was obtained in sealed bottles, and was described as "spectrally pure".

In the preliminary experiments hydrogen was introduced into the system by diffusion through a palladium tube, which was heated by a miniature electric furnace through which a slow stream of hydrogen was passed. Although this is one of the best methods of obtaining pure hydrogen, it is rather tedious for large volumes, and it was found more convenient to seal on to the pump a bottle which had been filled with hydrogen from an orthodox purification train. The gas was purified on the system before use by means of activated charcoal.

3. Procedure.

At the beginning of an experimental run with a new sample, the metal and absorption bulb were thoroughly degassed by baking-out at a temperature higher than that to be used in any subsequent solubility determinations. The rate of gas evolution from any part of the apparatus could be readily checked by carrying out a test collection in the gas-analysis system. When this cut-off test indicated that degassing was sufficiently complete, the value of the free gas space (or dead space) in the absorption bulb was determined by the admission of neon. The calibration was made at several temperatures and the temperature coefficient determined, so that appropriate correction for small temperature fluctuations could be applied to the data subsequently obtained with hydrogen. Because of the low solubility and rate of diffusion of neon in both the boro-silicate glass and silica, no corrections for these effects were necessary.

When the silica absorption bulb was used, two different methods were adopted in order to determine the true hydrogen-absorption curves. In the first procedure, instead of introducing hydrogen into the outer jacket to oppose the permeation of gas from the inner bulb, the jacket was continuously evacuated and the hydrogen diffusing out was collected in the analytical system of the pump table. The advantage of this method was that precise correction could then be made for both solution of hydrogen in the silica, and permeation through the wall of the bulb. Typical data obtained are shown in Fig. 2, in which are plotted the initial readings for (a) the total absorption of hydrogen by the sample and its surroundings, and (b) the cumulative volume of hydrogen diffusing through the silica and collected in the analytical system. As will be seen from curve (b) a certain period of time elapses

before the steady (linear) permeation condition is established, i.e. before the full concentration gradient of hydrogen is set up through the bulb wall. This time lag L , defined by the point at which the

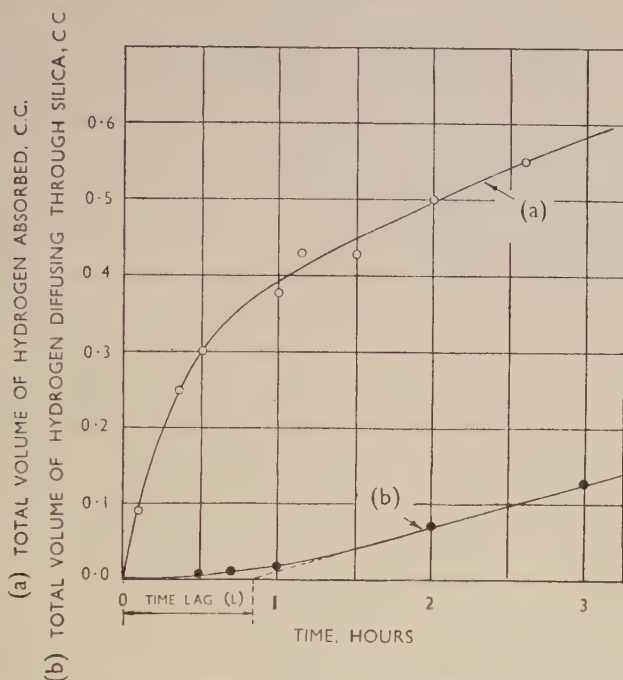


FIG. 2.—Initial Readings of an Experiment with Silica Apparatus, with no Hydrogen in Outer Jacket.

linear portion of the curve, produced backwards, intersects the time axis, is given by:¹²

$$L = d^2/6D \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

where d is the thickness of the silica wall and D is the diffusion constant ($\text{cm}^2 \text{sec}^{-1}$) of hydrogen in silica.

The steady rate of permeation P ($\text{cm}^3 \text{sec}^{-1}$), determined from the slope of the linear portion is:

$$P = ADC/d \quad . \quad . \quad . \quad . \quad . \quad . \quad (2)$$

where A is the area of the bulb and C is the concentration of hydrogen in solution at the inner surface of the bulb; the concentration at the outer face is assumed to be zero. As d and A could be determined by direct measurement after the experiment, it was possible to

calculate C , and hence the volume of hydrogen dissolved in the silica, since:

$$\text{Total hydrogen dissolved} = ACd/2.$$

In the second procedure the pressure of hydrogen in the inner bulb was opposed by an equal pressure in the outer jacket. The hydrogen was admitted to the outer jacket while the inner bulb was still being pumped, and sufficient time was allowed for the hydrogen-concentration gradient to be established from the outside to the inside of the bulb. The total volume of hydrogen dissolving in the wall from the inside when hydrogen was now admitted to the sample was less than if the gas had been admitted simultaneously to both vessels. The correction to be applied for the hydrogen lost by solution in the glass under these circumstances could be calculated from the data determined in the earlier experiments in which the first procedure was used.

4. Results.

It was soon apparent that the rate of solution of hydrogen in the metal was very slow and that saturation values could be obtained only after exposures of several hours' duration. The slow rate of approach to equilibrium in experiments of this type is undoubtedly due to the high impedance of the oxide film on the free surface of the metal. It is impossible to avoid this film, even under the most favourable vacuum conditions, since in addition to the inevitable oxide present on the sample as introduced into the system, the metal is also exposed to the oxidizing gases evolved from the system during the initial baking-out operations. The importance of this oxide impedance was emphasized in an early experiment (No. 2 in Table I) in which the first absorption run showed a very slow rate of solution. In the interval before a second run could be carried out, the metal was accidentally allowed to freeze, and on increasing the temperature again the crucible cracked badly along its whole length, but the molten metal was fortunately still retained. The freshly exposed surface, presumably free from oxide, resulted in a much enhanced rate of solution in the second measurement and equilibrium was attained in a much shorter time than in any of the subsequent experiments.

The results of the various measurements are summarized in Table I. The times given in the Table are those required for saturation to be reached. Considerably longer times were allowed in several of the experiments; it was found, however, that in experiments 7 and 8 a slight but continuous drift in the readings took place when very long exposures were used. Fig. 3 is a typical absorption curve obtained in

TABLE I.—Summary of Liquid Solubility Determinations.

Experiment No.	Temp., ° C.	Time, hr.	Absorption of Hydrogen, c.c./100 g. metal at 760 mm. pressure.			
			Total Absorption.	Solution in SiO ₂ .	Permeation through SiO ₂ .	Corrected Solubility.
2	670°	2	0.70	Nil	Nil	0.7 ₀
3	710°	8	1.39	0.16	0.26	0.9 ₇
4	750°	5	1.81	0.16	0.56	1.0 ₉
7 (a)	850°	3½	1.92	0.12	Nil	1.8 ₀
(b)	730°	...	1.30	0.12	Nil	1.3 ₀
(c)	790°	12	1.73	0.12	Nil	1.6 ₁
(d)	725°	16	1.01	0.12	Nil	0.8 ₉
8 (a)	730°	14	1.08	0.10	Nil	0.9 ₈
(b)	750°	12	1.50	0.10	Nil	1.4 ₀
(c)	820°	9	2.30	0.10	Nil	2.2 ₀
(d)	700°	14	1.20	0.10	Nil	1.1 ₀

Experiment 2: Boro-silicate glass bulb (split crucible).

Experiments 3 and 4: Silica bulb with outer jacket evacuated.

Experiments 7 and 8: Silica bulb with hydrogen in outer jacket.

Experiment 7 (b): The sample, after saturation at 850° C. (7 (a)) was cooled to 730° C. and held there for 40 min.

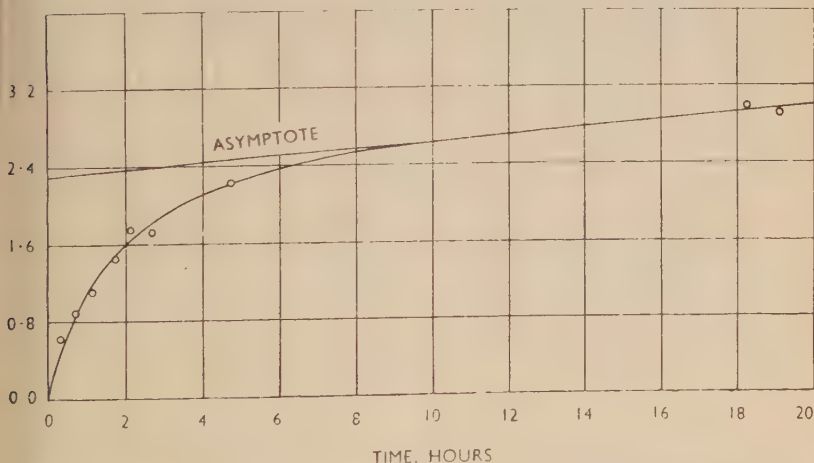


FIG. 3.—Solution of Hydrogen in Liquid Metal. Hydrogen absorption curve in experiment 8 (c).

one of these experiments. This drift appeared to approach a constant value, and the results in Table I have been corrected by deducting the asymptotic value of this error. The cause of this drift is obscure; it may possibly be due to solution of hydrogen in the tap lubricants.

By analogy with other similar gas-metal systems the logarithm of the solubility should be a linear function of the reciprocal of the absolute temperature. The results in Table I have been plotted on this basis in Fig. 4. The scatter is rather greater than it had been hoped to achieve, but in view of the inherent difficulties of the method, it is not un-

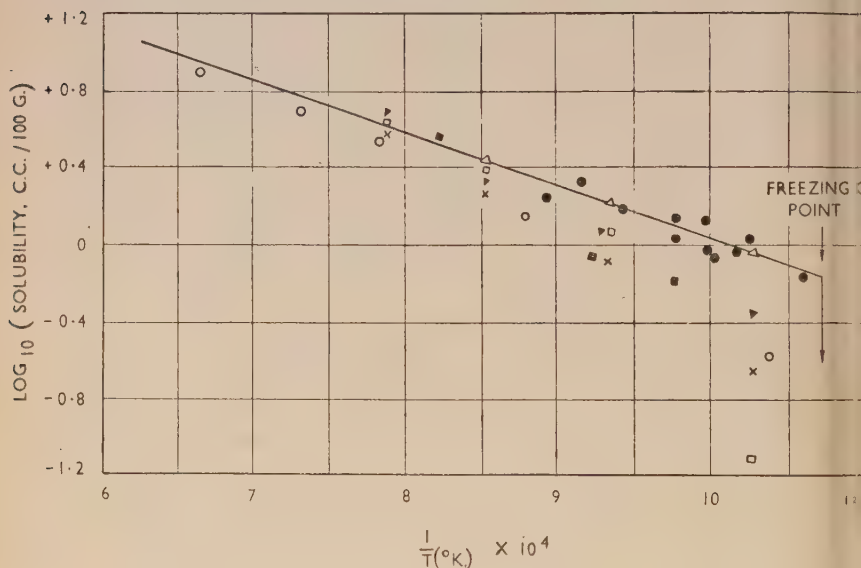


FIG. 4.— $\log S \sim \frac{1}{T}$ Plot of all Data on Solubility in Liquid Metal.

□ Rontgen and Braun, 1932.
○ Rontgen and Möller, 1934.
× Bircumshaw, 1935.
△ Baukloh and Oesterlen, 1938.

KEY.

■ Winterhager, 1939.
▼ Baukloh and Redjali, 1942.
● Present work.

reasonable. The best straight line through the data is represented by the following equation:

$$\log_{10} S_0 = -\frac{2760}{T} + 2.796 \quad . \quad . \quad . \quad (3)$$

where S_0 is the solubility at 1 atm. pressure and T is the absolute temperature.

If the solubility is assumed to vary as the square root of the pressure, this equation may be written:

$$\log_{10} S/p^{\frac{1}{2}} = -\frac{2760}{T} + 1.356 \quad . \quad . \quad . \quad (4)$$

where p is the pressure of hydrogen in mm. of mercury.

The solubility values found by previous investigators are also plotted in Fig. 4. This latter plot indicates the tendency of the earlier data to converge at higher temperatures and to show an increasing departure from linearity at low temperatures. As stated earlier in this paper, it is considered that these low results are due to the fact that usually the time allowed for solution was not sufficient for equilibrium to be attained. It will be noted that the data of Baukloh and Oesterlen (1938) are in exact agreement with the present results, and that the later results of Baukloh and Redjali (1942) appear to be too low.

III.—THE SOLUBILITY OF HYDROGEN IN SOLID ALUMINIUM.

1. *Apparatus.*

Fig. 5 is a sketch of the apparatus which was constructed for measuring the solubility of hydrogen in solid aluminium. The tube inserted in the furnace was made of the special boro-silicate glass, and the remainder of the apparatus shown in Fig. 5 in hard glass, which joins directly to it. The only part of the assembly which calls for special comment is the boat. This consisted of a rectangular frame of the special glass, fitted with prongs along each side, which scraped against the inside of the tube; a long rod resting along the bottom of the tube connected this frame to a small sealed-off U-tube containing a length of iron rod in each limb. With a powerful horse-shoe magnet the whole boat could be moved along the tube and, when necessary, rotated. It was thus possible to pick up a cylindrical metal specimen lying in the tube, to move it to and fro, or to dump it in a given position and slide the boat over the top of it.

The system shown in Fig. 5 was connected via a large-bore tap (lubricated with Apiezon grease) to the fore pump of the low-pressure gas-analysis table. Hydrogen could be admitted to the system through the palladium tube, and the pressure regulated and measured by means of the compression burette indicated in the sketch.

The temperature of the furnace was controlled by an energy regulator to $\pm 5^{\circ}\text{C.}$, and was measured by means of a thermocouple tied to the outside of the tube. The actual temperature of specimens under the normal conditions of heating was checked in a separate experiment by means of a second couple inserted in a hole drilled in a dummy specimen. It was found that the temperature of the specimens was approximately 5°C. lower than indicated by the external couple, and due correction was made for this error.

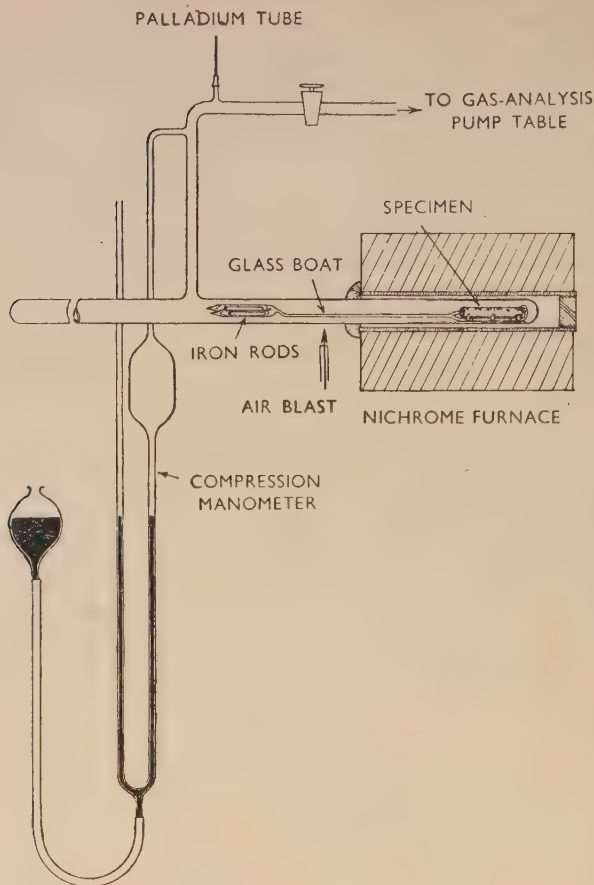


FIG. 5.—Apparatus for Measurements on Solid Metal.

2. Materials.

A standard size of specimens was used for all the measurements. This consisted of a cylinder, approximately 12 mm. dia. and 60 mm. long, weighing about 18 g. In all, five specimens were used, which were prepared from the following materials:

(a) Super-purity aluminium from the same batch as that used in the liquid solubility experiments. The metal was melted *in vacuo* in a graphite crucible by means of a high-frequency induction set, and maintained at 800°–850° C. for 3–4 hr. By slowly raising the heater coil, the metal was frozen in the crucible from the base upwards; a

completely sound rod 1 in. dia. and 4 in. long was obtained in this way, from which the specimen was turned. This specimen was coarse-grained, most of the crystals being several millimetres in diameter.

(b) A rod of vacuum-melted metal, similar to (a), which was grown into large crystals by straining 2% and annealing at 625° C. A standard cylindrical specimen was obtained which consisted of four crystals only.

(c) A section of super-purity ingot, again of the same batch as (a), but not melted in a vacuum, which was rod-rolled to give 75% reduction in area. A standard specimen was turned from the cold-worked metal and annealed by heating slowly to 500° C. in the solubility apparatus. After the experiments, this specimen had a grain-size of approximately 6 grains/mm.²

(d) A sample of cast commercial-purity aluminium. The porosity of this metal was nil by density measurements, and it appeared quite sound under the microscope. The grain-size was about 50 grains/mm.² and did not change appreciably during the solubility measurements. The analysis of the metal was :

Si, %.	Fe, %.	Cu, %.	Mn, %.	Na, %.
0.25	0.33	0.007	0.009	<0.0003

(e) Another sample of cast commercial-purity metal similar to (d), again with nil porosity. The grain-size was rather large (0.3 grains/mm.²), and the analysis of the metal was :

Si, %.	Fe, %.	Cu, %.	Mn, %.	Na, %.
0.23	0.33	0.007	0.006	<0.0003

3. Procedure.

Before each measurement the tube and boat were thoroughly degassed by heating to 650°–670° C., and the remainder of the apparatus where possible was baked out with a hand-torch. The specimen was then degassed at the maximum temperature permitted by the special conditions of the individual experiments; this was normally 625° C., but in some experiments in which attempts were made to keep the grain-size small, it was as low as 500° C. When the rate of gas evolution had been reduced to negligible proportions, the boat and specimen were withdrawn from the furnace. The large tap leading to the gas-analysis apparatus was now closed and hydrogen admitted to the system *via* the palladium tube. When the pressure and furnace temperature had been adjusted to the required values, the specimen was re-inserted in the furnace and saturated with hydrogen. The time necessary for equilibrium to be attained was assessed from data on the rate of degassing

at various temperatures. The time actually allowed was usually greatly in excess of this calculated value.

After the exposure the specimen was chilled by withdrawing the boat quickly from the furnace into a zone where the tube was cooled by an air blast. With hydrogen still present in the apparatus this provided a sufficiently rapid quench to retain substantially all the gas

dissolved in the metal.

The hydrogen was now pumped away and the specimen tipped out of the boat, which was pushed back into the furnace. The tube and boat were baked out again at 650°–670° C., and the temperature reduced to that at which the specimen was to be degassed. A careful cut-off test was then carried out over a period of $\frac{1}{2}$ –1 hr., to determine the rate of evolution of gas from the tube and boat, before the specimen was picked up and pushed into the furnace. Since the volume of gas dissolved by the specimen was extremely small and the extraction of it took up to 3 hr., heating at the lower temperatures, this cut-off

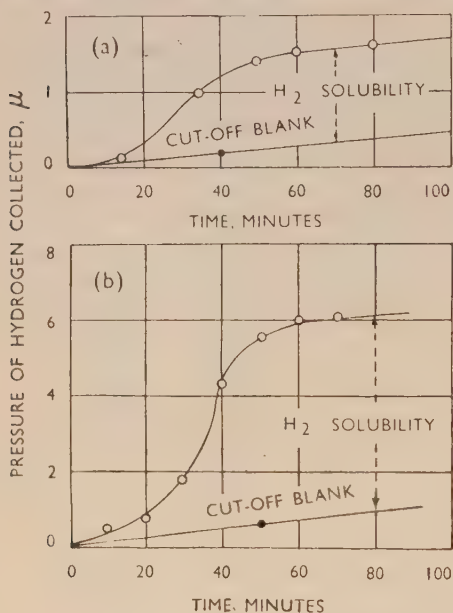


FIG. 6.—Typical Extraction Curves in Solid-Solubility Experiments.

- (a) Specimen (e) saturated at 545° C. in 88 mm. hydrogen pressure;
- (b) Specimen (d) saturated at 605° C. in 760 mm. hydrogen pressure.

blank was not always negligible, but had to be subtracted from the total gas collected.

Several determinations were made on each specimen. Two typical extraction curves, illustrating the order of the blank corrections, are shown in Figs. 6 (a) and 6 (b).

4. Results.

In the earlier experiments of the series, the apparatus had not been equipped with the compression manometer shown in Fig. 5, and hydrogen could only be admitted to slightly sub-atmospheric pressures.

because of the decreasing rate of diffusion through the palladium tube as the internal pressure approached atmospheric. The solubilities determined under these conditions were corrected to the value at one atmosphere by means of the $p^{\frac{1}{2}}$ law.

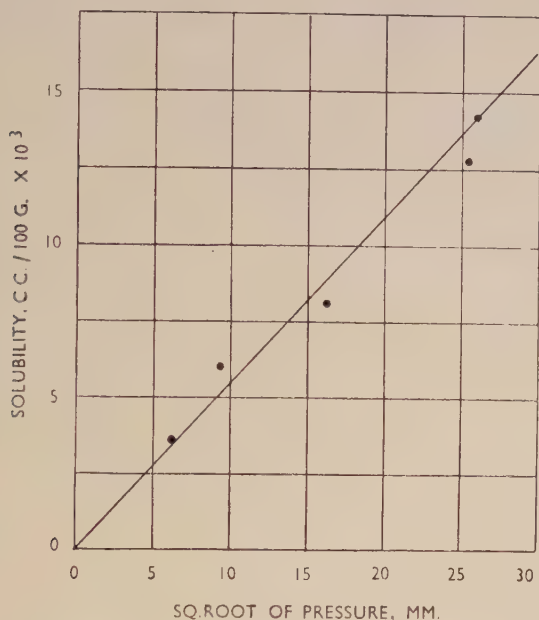


FIG. 7.—Variation of Solubility with Hydrogen Pressure (Solid Metal).

The validity of this law was subsequently established by some measurements on commercial-purity metal (sample (e)). These are summarized in Table II and plotted in Fig. 7. It is clear that the $p^{\frac{1}{2}}$ law is quite valid, and that all our measurements relate to a true solution of hydrogen in the metal.

TABLE II.—Validity of the $p^{\frac{1}{2}}$ Law. Commercial-Purity Sample (e). Tests at 545° C.

Hydrogen Pressure, p , mm.	$p^{\frac{1}{2}}$.	Solubility S , c.c./100 g.	$k = S/p^{\frac{1}{2}}$.
37	6.1	0.003 ₆	5.9×10^{-4}
88	9.4	0.006 ₀	6.4
262	16.2	0.008 ₁	5.0
640	25.3	0.012 ₈	5.1
675	25.9	0.014 ₂	5.7

The solubility determinations on the various specimens are summarized in Table III, which gives the details of the times allowed for saturation, and of the order in which the determinations were carried out.

TABLE III.—*Summary of Solid-Solubility Determinations.*

Specimen.	Temp., ° C.	Order of Determination.	Time, hr.	Solubility, c.c./100 g.
Vacuum-furnaced super-purity metal (a)	595°	3	18	$2.5_9 \times 10^{-2}$
	570°	1	89	2.1_9
	545°	2	28	1.8_4
Vacuum-furnaced super-purity metal (b)	620°	6	3	2.9_4
	595°	1	21	2.4_6
	565°	4	21	2.0_1
	535°	3	$39\frac{1}{2}$	1.4_9
	495°	2	70	1.3_0
	475°	5	71	1.0_2
Cold-rolled super-purity metal (c)	595°	5	$17\frac{1}{2}$	2.4_0
	580°	6	$18\frac{1}{2}$	2.4_4
	545°	2	$17\frac{1}{2}$	1.6_9
	520°	3	$19\frac{1}{2}$	1.3_6
	470°	1	45	1.1_9
	465°	4	38	1.0_0
Commercial-purity metal (d)	605°	...	$18\frac{1}{2}$	2.9_1
Commercial-purity metal (e)	545°	1.5_2^*

* Extrapolated from $p^{\frac{1}{2}}$ curve (Fig. 7).

The results given in Table III are plotted in Fig. 8 as the logarithm of the solubility *versus* the reciprocal of the absolute temperature. When the smallness of the hydrogen contents is considered, the results are very satisfactory. The points fall quite closely on the straight line represented by the equation:

$$\log_{10} S_0 = -\frac{2080}{T} + 0.788 \quad . \quad . \quad . \quad (5)$$

where S_0 is the solubility at 1 atm. pressure and T is the absolute temperature, or:

$$\log_{10} S/p^{\frac{1}{2}} = -\frac{2080}{T} - 0.652 \quad . \quad . \quad . \quad (6)$$

where p is the pressure of hydrogen in mm. mercury. The extrapolated solubility at the melting point (660° C.) is 0.036 c.c.

The results for the commercial metal are in sufficiently good agreement with those for the super-purity material to suggest that the true

solubility does not alter appreciably with the variations in purity within the normal limits.

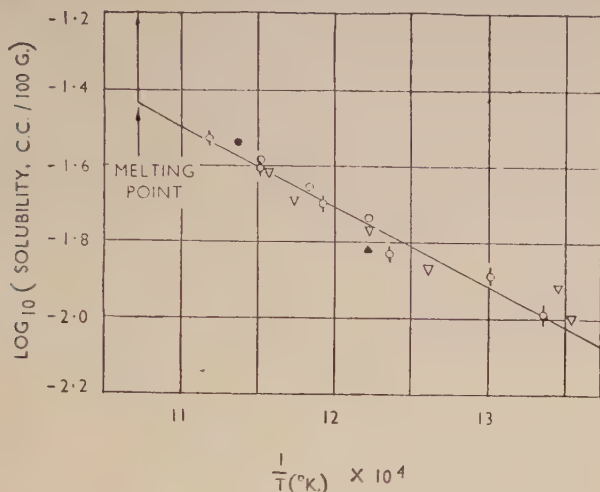


FIG. 8.—Solubility of Hydrogen in Solid Aluminium.

KEY				
○	Super-purity specimen (a).	●	Commercial-purity specimen (d).	
○	” ” ” (b).	▲	” ” ” (e).	
▽	” ” ” (c).			

IV.—THE RELATION BETWEEN THE HYDROGEN CONTENT AND THE POROSITY OF CAST TEST BARS.

The problem of “pin-holing” in aluminium and its alloys has been the subject of a very large number of papers in the scientific and technical journals over the last twenty or thirty years. Recent work has now made it fairly clear that the defect is caused entirely by hydrogen, which is rejected by the metal during solidification as a result of the marked change in solubility at the freezing point. It has been suggested, indeed, that this conclusion is an over-simplification, and ignores the possibly important roles which may be played by other elements, such as nitrogen and carbon. There is no experimental evidence, however, to indicate that the formation of compound gases, which is sometimes a cause of porosity in other metals, is a factor of any importance in the case of aluminium. Hydrogen, in fact, appears to be the only gaseous element which is appreciably soluble in the metal in the temperature range normally considered.

It is not intended to discuss this problem in any detail in the present paper. But in view of the data on the solubility of hydrogen in solid aluminium presented in Section III, it is of interest to consider what quantitative relationship exists between the hydrogen content and the porosity of metal cast under specified conditions. Metcalfe¹¹ has carried out some measurements on these lines. He found that the porosities of standard test bars cast from a 4.6% copper alloy, when plotted against the hydrogen contents as determined by vacuum fusion, gave a reasonably straight line. This line was extrapolated to zero porosity to give a gas-content value of 0.09 c.c./100 g., and this was taken to represent the probable solid solubility of hydrogen in the metal.

We have made measurements of a similar nature on a large number of bars cast from commercial-purity aluminium. These bars were cast in pairs in a skin-dried sand mould, and were run horizontally with adequate feeding. Every effort was made to ensure constancy in the casting conditions. Although several batches of metal were used in the tests the composition did not vary to any important extent from melt to melt, the principal impurities being iron (0.45%) and silicon (0.2%). The porosities of the bars were determined with considerable care, and the results expressed in the form :

$$\text{Percentage Porosity} = \frac{(\text{Theoretical Density}) - (\text{Actual Density})}{(\text{Theoretical Density})} \times 100.$$

The hydrogen contents were determined on samples 10 mm. dia. and 40 mm. long, which were turned from the bars. The vacuum-extraction procedure has been described in an earlier paper,¹⁰ but it should be mentioned that in all the present measurements the gas was extracted from the specimens at 600° C., i.e. in the solid state.

In Fig. 9 the percentage porosities of the bars have been plotted against their true hydrogen contents. There is a considerable scatter in the points, but a highly significant correlation exists between porosity and the hydrogen content, and the best line which can be drawn indicates that :

(a) No porosity occurs until the hydrogen content is greater than 0.12 c.c./100 g.

(b) The linear increase in porosity with increase of hydrogen content above this value is such that 1.0 c.c. of porosity (i.e. 1%) is produced by 0.46 c.c. of hydrogen (measured at room temperature). The pressure in the pores at the time of their formation can therefore be assessed to be between 1 and 1½ atm., the actual value depending on the final temperature of solidification.

The discrepancy between the equilibrium solid solubility of hydrogen in the pure metal (0.036 c.c.) and the value of 0.12 found from the porosity relationship can be ascribed to a supersaturation effect. The linear character of the relationship indicates, however, that any such effect must be independent of the initial concentration of gas in the metal. A consideration of the solidification process shows that this is quite feasible. The rapid rate of diffusion of hydrogen in the solid metal at these temperatures makes it very probable that in the early stages of freezing there will be an equilibrium partition of gas between the solid and liquid present in the partially solidified mass. The concentration of gas in the liquid therefore rises rapidly as the solidification proceeds, and when it exceeds the saturated solubility (by an amount which will be determined by nucleation conditions) hydrogen will be precipitated in the form of bubbles. Once this separation of gas has been initiated, the "internal pressure" of hydrogen in solution will remain constant, i.e. the concentration of hydrogen in the final liquid will be independent, over quite wide limits, of the original hydrogen content of the metal. As the solidification of the metal is completed there will be a strong tendency for the hydrogen dissolved in this final liquid to be retained in solution, especially if the liquid is of eutectic composition, and thus to produce the constant supersaturation effect indicated by the porosity data.

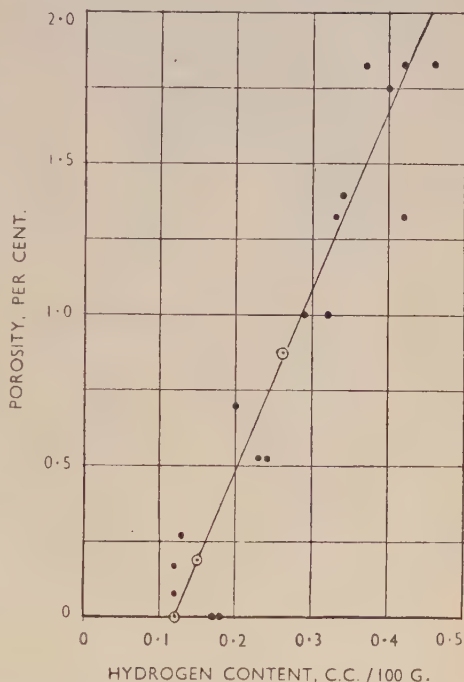


Fig. 9.—Relation Between Hydrogen Content and Porosity in Sand-Cast Bars of Commercial-Purity Aluminium.

KEY.

- Single determinations.
- Mean of two or more determinations.

V.—DISCUSSION.

From the experimental work reported here, the mean values of solubility given in Table IV are derived. The extrapolated value of solid solubility at 0° C. is included only as an indication of the probable order of solubility at this temperature.

TABLE IV.—*Mean Values of Solubility at 1 Atm. Pressure.*

Temp., ° C.	Solubility, c.c./100 g.	Temp., ° C.	Solubility, c.c./100 g.
0°	1×10^{-7}	660° (liquid)	0.6 ₉
300°	0.001	700°	0.9 ₂
400°	0.005	725°	1.0 ₇
500°	0.012 ₅	750°	1.2 ₃
600°	0.026	800°	1.6 ₇
660° (solid)	0.036	850°	2.1 ₅

The solution of hydrogen in aluminium can be represented by the equation :



and the equilibrium constant K of the reaction may then be written :

$$K = (\text{H})^2 / (p\text{H}_2)$$

where H is the concentration of hydrogen in the metal and $p\text{H}_2$ the pressure. It follows that :

$$\frac{d \log_e K}{dT} = \frac{\Delta H}{RT^2}$$

ΔH is the heat of solution of one gram molecule of hydrogen. Thus we have :

$$2 \log_e S_0 = -\frac{\Delta H}{RT} + c$$

where S_0 is the solubility and c a constant.

The heat of solution is therefore given by :

$$\Delta H = 2.303(2RA) = 9.14A$$

where A is the slope of the plot of $\log_{10} S$ against $\frac{1}{T}$.

The values of ΔH which we find for liquid and solid aluminium are given in Table V. Similar data can be calculated for three other metals, and these are also given in the Table. It will be seen that the change in ΔH in passing from the liquid to the solid state is comparatively small for all four elements.

The solubility of hydrogen in liquid aluminium is much higher than has previously been assumed, since the results of Baukloh and Oesterlen

have generally been disregarded because of the weight of other evidence. For example, the somewhat speculative deductions of Dardel,¹³ in a recent paper, are based on solubility values which are too low.

TABLE V.—*Heats of Solution of Hydrogen in Liquid and Solid Metals.*

Metal.	ΔH in cal./g.mol.	
	Liquid.	Solid.
Aluminium	25,000	19,000
Copper	23,800	27,900
Iron	15,180	13,300 (α)
		13,100 (γ)
Nickel	8,600	5,900

We have found that the volume of hydrogen dissolved in commercial melts of aluminium and its common alloys is usually of the order of 0.1–0.6 c.c./100 g. It is instructive to consider this range of values in terms of the equivalent “internal pressures” indicated by the solubility data. For example, a melt of aluminium maintained at 725° C. and containing 0.3–0.4 c.c./100 g. of hydrogen, will have an “internal pressure” of about 0.1 atm.; if the gas content is reduced to 0.1 c.c., the corresponding value will fall to 0.01 atm. This means that if the metal is degassed by passing a stream of bubbles of neutral gas through it, the theoretical maximum rate of removal of hydrogen will initially be 10% of the volume of gas passed, but this will fall to 1% as the hydrogen content approaches 0.1 c.c. Because of the limited time of contact of the bubbles with the melt, and the operation of other factors tending to restrict the diffusion of hydrogen into them, it is very unlikely that the percentage of hydrogen carried out of the metal even approaches the theoretical value. The practical difficulty of reducing the hydrogen content to a low level by this means is thus readily understood.

The solubility of hydrogen in the solid metal is so small that it would be possible for the bulk of the gas to be accommodated as a monatomic film at the grain boundaries. It was for this reason that the experiments were devised to establish whether the solubility is a function of grain-size. The results indicate that it is not, and this is to be expected by analogy with other metals.

The results given in Section IV of the paper are of some immediate practical interest. The data that we have available, however, are not sufficient to permit of a more quantitative treatment of the general

problem of pin-holing. The mode of freezing of commercial-purity aluminium is rather too complex to permit of a detailed analysis of the process of separation of hydrogen in our sand-cast bars. A rough assessment of the *maximum* retention of gas by the solid can be made if it is assumed that the main eutectic arrest is at 655° C. (Al-FeAl₃, at 1.8% Fe). At this temperature there will be approximately 25% of liquid, and the volume of hydrogen which can still be retained in solution in liquid and solid metal (in equilibrium with 1 atm. pressure) will be :

$$(0.66 \times \frac{25}{100}) \text{ c.c./100 g. in the liquid}$$

$$(0.035 \times \frac{75}{100}) \text{ c.c./100 g. in the solid}$$

or a total of 0.19 c.c./100 g. It is assumed that any hydrogen in excess of this value will have previously separated as bubbles during the cooling from 660° to 655° C. If solidification were now to be completed at 655° C. it would be possible for most of this hydrogen to be chilled into supersaturated solid solution. In fact, of course, an appreciable amount of liquid persists down to the ternary eutectic temperature (577° C.), so that this value of 0.19 c.c./100 g. is certainly an over-estimate, and the experimental value of 0.12 c.c./100 g. appears very plausible.

If the main separation of bubbles occurs at about 655° C., and a suitable correction is made for the contraction of the metal during solidification and cooling to room temperature, the pressure in the pores at the moment of their formation is calculated to be about 1.3 atm.

It would be very useful if similar measurements of porosities and hydrogen contents could be made on a range of simple alloys prepared from high-purity materials. If such data were available, it should be possible to develop a fairly clear picture of the mechanism of porosity formation in aluminium castings.

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CORRESPONDENCE ON PAPER BY DR. PAUL
LACOMBE AND M. LOUIS BEAUJARD:
"THE APPLICATION OF ETCH-FIGURES
ON PURE ALUMINIUM (99.99%) TO THE
STUDY OF SOME MICROGRAPHIC
PROBLEMS."

(*J. Inst. Metals*, this volume, p. 1.)

DR. U. R. EVANS,* M.A. (Member): I would express my sincere congratulations to both authors for their admirable work, some of which I had the privilege of seeing during a recent visit to Vitry. The results obtained by their new micrographic method will greatly increase our understanding of the structure of metals.

Three points appear to be brought out clearly:

(1) Special attack at the grain boundaries has been obtained on aluminium of *very high purity*, where it is impossible to attribute the localization of corrosion either to the presence of some particularly attackable impurity, or to impoverishment in some ennobling constituent.

(2) It is indicated that the attack at the common boundary between two adjacent grains increases with *increasing difference in their orientation*. This point is so important that it would be very welcome if the authors could print a series of photomicrographs, etched under identical conditions, embodying cases where the relative orientation of neighbouring grains varies over a range of angles, and bringing out the fact that the depth of the trenches increases steadily with the angular difference. If a quantitative relationship between the angle and the depth of attack could be established, this would greatly aid our understanding of the subject and enable it to be placed on a mathematical basis. Meanwhile there is no difficulty in accepting and interpreting the facts. It is indeed to be expected that the energy needed to dig out an atom from the centre of the grain, where it is surrounded by other atoms in perfect crystal array, will be greater than that needed to dislodge an atom situated at a grain boundary where the orientation on two sides is different; also that at a grain boundary the energy needed will decline steadily as the angular difference between the two orientations increases. Thus, the removal of an atom will be a more frequent occurrence at the boundary between the two grains of greatly differing orientation than at the boundary between grains of similar orientation.

(3) The fact that individual slip lines can be traced across the grain boundary shows, as the authors point out, that gliding on one grain is not independent of gliding on its neighbours. The slight irregularities to be observed in the slip planes where they approach the boundaries bring out, however, the fact that in this region the deformation process is of a different character and doubtless absorbs more energy than that proceeding in the centre of the grains. In fine-grained metal these barriers, where extra energy-uptake will be needed for deformation, are more frequently encountered than in coarse-grained metal; and this goes far to explain why, especially

* Cambridge University.

in non-cubic metals, fine-grained material is harder than coarse-grained material.

DR. W. BOAS * : I have read the paper with very great interest and would like to congratulate the authors on their beautiful technique. There are three remarks which I want to make, all of which confirm the authors' observations.

There is considerable evidence for the conclusion drawn in the paper that the deformation of a crystal depends on that of its neighbours. A recent investigation † has shown that the deformation of a crystal in the neighbourhood of its boundary may be larger or smaller than at its centre according to whether its neighbour was, on the average, more or less deformed than the crystal under consideration.

The second remark refers to the depth of distortion produced by mechanical polishing (p. 3). An X-ray investigation ‡ carried out on very similar lines to that referred to by the authors showed the same depth of deformed transition zone for crystals of aluminium and copper (30 and 20 μ , respectively); however, for crystals of the non-cubic metals zinc, cadmium, and antimony this depth was found to be about 5–10 times greater, the reason for the difference probably being the greater number of slip systems in the cubic metals.

Finally, I was very interested in the multiplicity of grain boundaries obtained after one prolonged annealing (p. 14). References to a similar effect were made by Vogel,§ and by Jones || on so-called "abnormal structures" in lead. About two years ago, Miss V. Hawkins ¶ observed such multiple "grain boundaries" in highest-purity aluminium in the course of an investigation of grain growth on annealing of recrystallized aluminium. The investigation has not been published since we came to no conclusion as to the nature of the network; however, since our evidence goes further than that of the authors we may be allowed to give some of our results.

The network was observed on etching the specimens which had been deformed by compression, annealed at 500° C. for 1 hr., polished electrolytically, and then annealed for a long period. Care was taken to avoid any strain due to a temperature gradient in the specimens, although it was shown that the rates of heating and cooling had no effect on the development of the multiple boundaries. In some specimens the network was already detectable before etching. Etching before the prolonged anneal did not reveal the network, which therefore could not have been an etching effect, but must have been a consequence of the annealing treatment.

The network was a surface effect. If a surface showing the network was polished electrolytically and re-etched, no network was revealed. Neither did it become visible when such a specimen was cut and the new surface polished and etched. In the former case, only that set of lines was developed which corresponded to the final position of the boundaries.

No multiple boundaries were observed in specimens of commercial aluminium. Their occurrence was restricted to specimens of high-purity aluminium. In these specimens not all the boundaries became multiple,

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† W. Boas and M. E. Hargreaves, *Proc. Roy. Soc.*, 1948, [A], **193**, (1032), 89.

‡ W. Boas and E. Schmid, *Naturwiss.*, 1932, **20**, 416.

§ R. Vogel, *Z. anorg. Chem.*, 1923, **126**, 1.

|| B. Jones, *J. Inst. Metals*, 1937, **60**, 187.

¶ Now Metallurgy Dept., University of Sheffield. Miss Hawkins kindly allowed me to make the reference to her work done in Melbourne.

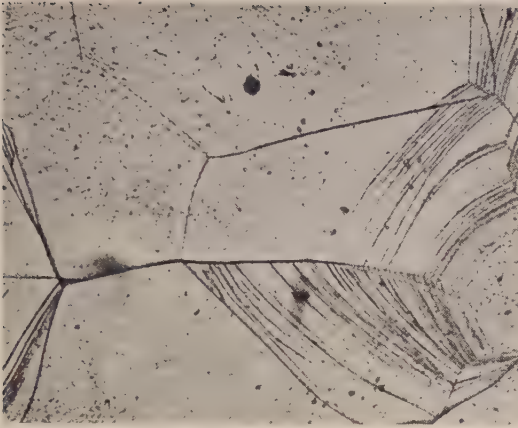


FIG. A.—Multiple Network of Grain Boundaries on the Surface of Pure Aluminium After Annealing at 450° C. for 1 Week. $\times 170$.



FIG. B.—Discontinuous Lines on the Surface of Pure Aluminium After Annealing at 450° C. for 1 Week. The glass tube containing the specimen was sealed off after being evacuated continuously for the first 24 hr. of the annealing. $\times 580$.

[To face p. 622.]

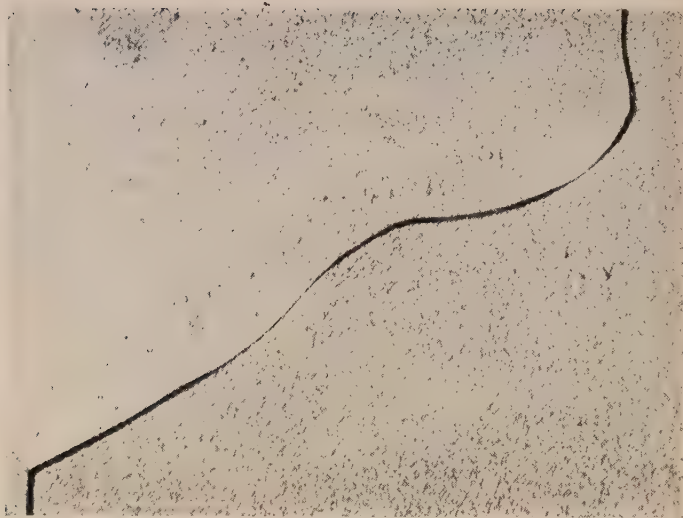


FIG. C. $\times 10$.

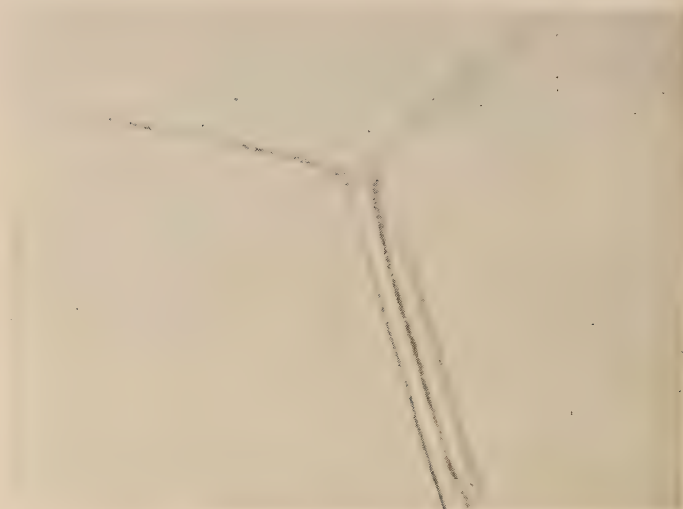


FIG. D. $\times 1500$.

the number of boundaries affected and also the number of intermediate lines depending largely on the period and temperature of the annealing treatment. No multiple boundaries were observed after annealing at 300° and 350° C. for 1 week, nor after annealing at 400° and 420° C. for 1 day. Several boundaries were observed after annealing at 400° and 420° C. when the annealing period was extended to 1 week. A few multiple boundaries appeared after annealing at 450° C. for 1 day, and after annealing for 1 week at this temperature they were present in abundance (Fig. A, Plate XLVI). If the annealing treatment was carried out at 550° C. many multiple grain boundaries were developed after as short a period as 3 hr. These observations show the influence of the temperature of annealing on the rate of appearance of the multiple boundaries.

In order to find out why lines which no longer represented the grain boundaries were brought out on etching, experiments were carried out under various conditions. Networks were observed after annealing at 450° C. for 1 week in all the following conditions, no differences in the appearance of the network being apparent: (1) annealing in a muffle furnace with free access of air, (2) annealing *in vacuo* (approximately 2×10^{-5} mm. mercury pressure of air), (3) annealing in oxygen-free dry hydrogen at approximately 1 atm. pressure (at room temperature) after the water vapour on the glass walls had been expelled by heating the glass to 420° C. for $\frac{1}{2}$ hr. under continuous pumping.

In the last two cases the specimens were sealed in Pyrex glass tubes. It was observed that gases were liberated from the metal during the annealing, and since the same effect occurred with mechanically polished specimens it was concluded that the gases were absorbed during the production of the metal and not during the electrolytic polishing.

In order to determine the effect of these gases on the development of the multiple network, glass tubes containing the specimens were continuously evacuated during 5 hr. of heating from room temperature to 450° C. and during the following 19 hr. of annealing at 450° C. Gases were liberated when the temperature exceeded 300° C. and for some time after 450° C. had been reached, but the liberation ceased after about 5 hr., and the pressure was below 10^{-2} mm. when the tubes were sealed off. The pressure was still the same after the subsequent annealing for 6 days. Multiple networks developed after this annealing treatment. However, there was a significant difference in their appearance in this case. The lines developed in the specimens annealed after the gases liberated had been pumped away, were markedly discontinuous (Fig. B, Plate XLVI). Indications of this had already been observed in the specimens annealed in hydrogen.

An explanation of the multiple boundaries can perhaps be given on the following lines. The boundaries move in steps and remain in certain positions for some time, thus allowing a reaction to occur between the metal and the annealing atmosphere. The reaction product is then revealed on etching. An alternative explanation is that the annealing atmosphere can only react with the metal during certain periods. For instance, the metal may be protected by a surface film in which stresses are produced when the grain boundaries move. If the stresses exceed a certain value, the film ruptures and the surface of the metal is free to react at the line of rupture. By repetition of this process, a number of such lines could be formed.

From the experimental evidence it cannot be decided which of these suggestions is correct, or whether a third explanation has to be found.

The AUTHORS (*in reply*): We should like first of all to thank Dr. Evans and Dr. Boas warmly for their interesting contributions to the discussion on our paper.

On the subject of the attack of high-purity aluminium at the grain boundaries, Dr. Evans points out how interesting it would be if it were possible to correlate quantitatively the speed of intergranular attack with the relative orientations of neighbouring grains. If the speed (or depth) of intercrystalline attack accelerated proportionally with an increasing difference in orientation of adjacent crystals, this would provide a further reason for attributing intercrystalline corrosion to the boundary structure itself rather than to impurities concentrated in it. In this connection one may recall the beautiful experiments of Chalmers * on a bicrystalline specimen of tin, whose tensile strength increased in proportion as the disorientation of the two crystals increased. Although we have not yet been able to apply the same experimental technique to the study of intercrystalline attack, in collaboration with Professor Chaudron and Dr. Yannaquis † we have recently obtained complementary qualitative results on the intercrystalline attack of high-purity aluminium by hydrochloric acid, and have shown, in particular, that the relative orientation of neighbouring grains is not in itself sufficient to determine whether their common boundary will resist attack or not. It is further necessary that the grain boundary should possess a particular orientation with reference to the two neighbouring networks. Thus, Fig. C (Plate XLVII) shows that a noticeable difference in speed of attack can exist along the length of one and the same winding boundary separating two randomly oriented crystals. It is remarkable that the unattacked regions should correspond to an almost identical average direction of the common boundary. Either side of the unattacked zones the crack due to intergranular attack widens, in proportion as the orientation deviates from a direction favourable to freedom from such attack.

Dr. Evans and Dr. Boas both agree that the deformation of a crystal depends on that of its neighbours, and we also have noted ‡ that a more intense distortion of the crystals exists in the region of their grain boundaries, as revealed by a deformation and rotation of the etch figures in relation to those observed at the centre of the crystals. It is probable, however, that this interaction of the crystals in the course of the deformation of the whole mass depends on the speed of deformation. In the case of very slow deformations (creep), one may agree with Kê § that the interaction is restricted to a minimum, the crystals shifting in a mass by a mechanism of viscous slip throughout the thickness of their grain boundary.

Finally, Dr. Boas directs attention to the appearance of multiple "grain boundaries" in the course of the same annealing treatment. He supplies data (based on the work of Miss Hawkins) on the conditions of appearance of this effect, particularly from the point of view of the purity of the metal, the nature of the annealing atmosphere and temperature. But in our opinion the most important point is to determine whether these multiple grain boundaries really indicate that the line of recrystallization advances by successive steps in the course of one and the same heat-treatment, contrary to the generally accepted idea of a continuous shifting of the grain boundary. We agree with Dr. Boas that these multiple boundaries may well be due to periodic ruptures of the surface oxide film above a certain limiting value of the stress. Moreover, we have proved, in as yet unpublished work, that on an electrolytically polished surface it is possible to reveal the displacement of a crystal grain boundary in process of growth, by a slight relief of the micrographic surface perpendicular to the grain boundary. In the case of a multiple

* B. Chalmers, *Proc. Roy. Soc.*, 1937, [A], **162**, 120.

† N. Yannaquis and P. Lacombe, *Compt. rend.*, 1948, **226**, 498; G. Chaudron, P. Lacombe, and N. Yannaquis, *Compt. rend.*, 1948, **226**, 1372.

‡ P. Lacombe and L. Beaujard, *Rev. Mét.*, 1947, **44**, 71.

§ T'ing-Sui Kê, *Phys. Rev.*, 1947, [ii], **71**, 533.

boundary, we have been able to show by examination at high magnification that each boundary corresponds to a different surface level. Fig. D (Plate XLVII) shows the displacement of a grain boundary in three stages. The photomicrograph has been taken by focusing on the central boundary, so that the boundaries on either side appear equally hazy. It is difficult to estimate the difference in level between these different planes, or to assess its importance, and only by a method of interferential microscopy such as Tolanski's might it be found possible to equate the total difference in level between two adjacent crystals, to a whole number of lattice parameters of the aluminium cube. Be that as it may, this experiment seems to us to afford yet one more decisive proof in support of the theory of a discontinuous recrystallization whose contours move in successive steps.

DISCUSSION ON PAPER BY DR. MAURICE COOK AND MR. EUSTACE C. LARKE: "CALCULATION OF LOADS INVOLVED IN METAL STRIP ROLLING." *

(*J. Inst. Metals*, this volume, p. 55.)

DR. HUGH FORD,† Wh.Sc., B.Sc.(Eng.): I was very glad to be invited to open this discussion, because it is the last act in a piece of co-operative work, which I think is so useful and productive in research. Present-day research frequently calls for such extensive facilities and staff that success can be achieved only by the co-operation of specialized groups. First of all the steel industry, the non-ferrous industry, and the makers of rolling-mill plant co-operated in providing the equipment on which our experiments, referred to in the paper, were carried out. These industries also provided the money to pay our running expenses. Secondly, Dr. Cook provided a large supply of specially prepared high-conductivity copper strip so that we could carry out our rolling tests. Thirdly, we carried out the tests and handed them over to Dr. Cook and Mr. Larke, so that they could examine them in relation to this method which they have discovered; and now I am asked to open the discussion on their paper.

I should like first of all to comment on some of their opening remarks about existing methods of calculation. They say that the methods put forward by Von Kármán, Trinks, Tselikov, and others are in general only of limited application, and that they are very approximate. Our experiments have provided the means of comparing all these methods and Orowan's graphical methods with experimental results, and it is found that these methods can be made to give reliable figures. The present authors have already shown in a previous paper‡ that Ekelund's method gives close agreement with Orowan's more exact method if a certain factor, which they have calculated as 1.05 in the case of copper, is used. We have shown§ that this factor of 1.05, by which the values of roll force calculated by Ekelund's formula have to be increased to give the same values as those obtained by Orowan's, can be dispensed with if the yield-stress value used is that which is the true mean yield stress over the arc of contact; so that in actual fact what the present authors showed in their earlier paper was even more to the point than they at the time suspected.

We have examined Von Kármán's method, and we have also examined Orowan's homogeneous graphical method, and we have shown that over the range of cold-rolling conditions both methods give results which differ very little from Orowan's "exact" method. We have shown that, by the choice of suitable coefficients of friction, i.e. within the reasonable range of what one might expect those coefficients to be, they all give results which are in reasonable agreement with the experimental results over the fairly wide range of conditions which our experiments covered. I think, therefore, that it is possible by the judicious use of these methods to get a reasonable estimate of

* Discussion at the Annual General Meeting, London, 16 March 1948.

† British Iron and Steel Research Association, London.

‡ *J. Inst. Metals*, 1945, **71**, 557.

§ "Researches into the Deformation of Metals by Cold Rolling", *Inst. Mech. Eng. Preprint*, 1948, (Apr.).

roll force and torque. The authors go on to say that these methods depend on the determination of experimental curves of resistance to deformation, which may necessitate the consideration of the effect of inhomogeneous deformation. This is a matter of some importance, since Orowan believed that there was possibly some variation in the yield-stress curve, depending on the amount of inhomogeneity of deformation caused by the rolling process; but we have again examined this factor, and we find that from a practical point of view it is of little importance in calculation, and can be, I think, neglected in cold rolling.

The next point which I should like to make concerns the additional assumption which is made, namely that the lever arm is a constant fraction of the length of the arc of contact. The authors have given in this paper some Tables (Tables II-IV inclusive) taken from my original report, in which this fraction (referred to as β in the paper) was calculated from the experiments, and they have shown that β is very nearly constant. Mr. Bland and I further examined this matter and found that a much closer correlation was obtained by considering the effect of roll flattening. Using Hitchcock's formula, it is possible to express the distance from the plane of exit at which the roll force can be considered to act, as a fraction of the flattened arc of contact. Thus:

$$\beta = \frac{1}{2} \sqrt{\frac{R}{R'}} - (\frac{1}{2} - \beta') \sqrt{\frac{R'}{R}}$$

where $\beta = \frac{\text{lever arm}}{\text{length of contact arc}}$

R = roll radius.

R' = radius of flattened arc of contact, from Hitchcock's formula.

β' = distance of line of application of roll force from the exit plane divided by the length of the arc of contact.

= 0.48 for polished rolls, 0.43 for matte-finish rolls, with commercial rolling oils.

We have found that β' is much more nearly constant than β for the whole of the test data.

The trend which can be seen in particular in Table IV is, I think, not an experimental inaccuracy. It will be noticed that in the higher passes the value of the ratio falls. That is because the roll flattening becomes progressively greater, and this variation is largely eliminated in the values of β' as defined above. The lever arm, therefore, is a reasonably constant fraction of the arc of contact, but where the strip is very thin or the rolls are large in diameter, i.e. where roll flattening may be large, this fraction shows a gradual variation.

The method, as the authors have said, depends on a single energy curve being obtained; i.e. that irrespective of the number of passes, the energy necessary to cause a given deformation is the same. Our experimental results have shown that this is an approximation. It is true in the case of highly polished, smooth rolls with good lubrication, but with matte-finished rolls, there seems to be a number of separate curves, depending on the amount of reduction in the pass. It looks as though the method works satisfactorily from the figures, but I should like to suggest that the results for the very thin copper strip (the 0.05 in. material) should be examined to see to what extent the method is applicable there. It is important to remember that the method depends on these two assumptions, but what is needed is a method that works in practice; it does not matter how many assumptions are made. Actually in this method, the fact that it is necessary to have a few experimental values to start with, means that all the variations and irregularities are fed back into the basic curve. When, therefore, any particular rolling schedule is calculated from the basic curve, these slight discrepancies are evened out over the whole of the

data, and the result is probably a practical approximation to these rolling loads. After all, what we want is to be able to predict results within $\pm 20\%$. If we achieve that, we have something which is good enough for the working out of rolling schedules in the mill plant.

The method depends on having available a few values of roll force. There are few cases where roll-force meters have been fitted to mills; where these are fitted, it is much simpler to measure all the passes, and be sure of the result directly. In the majority of mills, it is quite impracticable to fit roll-force meters. So it is necessary to come back to methods of calculation of roll force such as those of Von Kármán, Trinks, or Orowan, or any of the other methods, to establish first of all a few values of roll force. Then I believe that this method can be applied and rapid calculations and estimations can be made of any rolling schedule required. The final answer will be found in the judicious combination of many of these methods to give the engineer as rapidly as possible the particular information which he wishes to obtain. In this connection it is my opinion that Dr. Cook and Mr. Larke have rendered the engineer a great service, and I wish to congratulate them on an excellent piece of work.

MR. W. C. F. HESSENBERG,* M.A. (Member) (contribution read by Mr. D. R. Bland): The authors are to be congratulated on so neatly side-stepping the difficulties associated with friction and roll flattening. Even the more complete methods of rolling-mill calculation require a minimum of experiment to determine a suitable value for the coefficient of friction, and the authors have evidently decided that a little more experiment would pay for itself by greatly reducing the amount of computation required. Inspection of Figs. 5-8 suggests that for the particular range of rolling conditions considered their decision is justified.

The authors make no mention of the effect of speed. At present there does not appear to be any method of calculation which takes this into account, although it is frequently too large to be ignored. In the experiments examined by the authors, the speed of rolling is relatively low. It may well be that for higher speeds the method is equally applicable, so long as the speed is kept constant. This is a point which would seem to need further investigation before the method can be more widely applied with confidence.

The ease with which load curves can be constructed by this method suggests a simple trial-and-error procedure for quickly setting up rational or constant-roll-force roll-pass schedules. A series of load-thickness curves is constructed, as shown in Fig. A, for material 0.1 in. thick at the start. Relatively few points are required to construct a nest of curves of sufficient accuracy for this purpose. Suppose it is required to reduce the strip to 0.05 in. in a series of passes of equal roll load. A point *A* at 0.05 in. and 25 tons is selected as a trial. By following a course parallel to the nearest curve from *A* down to the draft axis to point *B*, one finds the thickness of strip which, if reduced to 0.05 in., would give a roll force of 25 tons. The process is repeated by taking point *C* at 25 tons and vertically above *B* and following the curve down to *D*. Finally, one reaches point *G* at approximately 23 tons. In view of this lower roll force in the first pass, a new trial is made, starting again at 0.05 in. but with a slightly lower roll force than the 25 tons originally chosen. In this way, after a few trials a constant roll-pass schedule is quickly obtained.

MR. D. R. BLAND †: The authors say, on p. 56: "The increase in kinetic energy of the material, associated with the difference in exit speed of the strip

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† British Iron and Steel Research Association, London.

and the peripheral speed of the rolls, is negligible, and the values of these speeds have, therefore, been taken as identical.' I agree that the kinetic energy is negligible compared with the work of deformation; but, if this argument were valid, the kinetic energy between entry and exit would also be negligible, and the same argument could be used to show that the entry speed of the strip was equal to the exit speed, which is far from being the case.

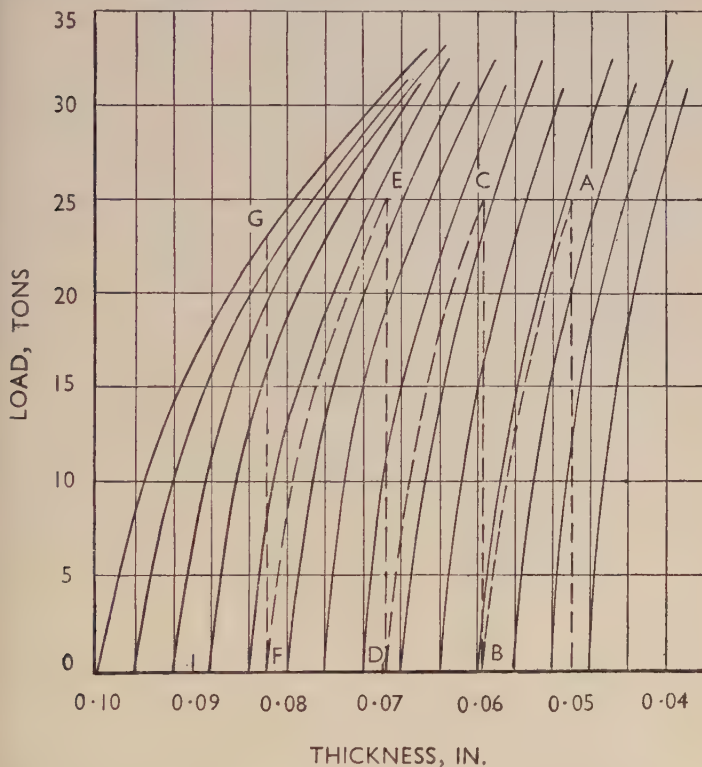


FIG. A.—Load-Thickness Curves for Material 0.10 in. Thick at Start.

Then comes the question of why that causes no error in this paper, and the answer is that the exit speed of the strip is very nearly equal to the speed of the rolls, but for a different reason from the one given here. The explanation is that the neutral point is very nearly at the exit, so that the forward slip is only of the order of about 1%, and the error by taking the two speeds as equal is also of the order of 1%. This assumption, however, would lead to very great errors indeed if a large front tension were applied with a forward slip of the order of 10%.

On p. 69 the authors say that the lack of constancy in the value of β causes the scattering of the points in the total-energy curve. I should be glad if they would explain why this is the case. I agree that it seems from the experimental results that the two occur at the same time, but I am not aware of any proof of why one should follow from the other.

PROFESSOR F. C. THOMPSON,* D.Met., M.Sc. (Member of Council): The two things which have struck me more than anything else about this paper are its simplicity of treatment and the ingenuity of the technique described. The remarkable accuracy with which the experimental results of, for instance, Fig. 2 fall on the smooth curve is convincing justification for the assumption which the authors have made, that the work done in effecting a given deformation is independent of the way in which that is carried out. That assumption is by no means self-evident, even in the case of a material such as copper, and it is even less so for a material such as steel, which may at times show marked strain-ageing; that confirmation by itself would have justified the publication of these results.

The next point of interest is the rather curious shape of the basic curves, as the authors call them, which show a kink, an arrest, or even a definite dip. The authors must have given some thought to this curious shape, and if they have any ideas on the subject, however tentative, they would be of great interest. One cannot but be struck by a possible analogy between the authors' curves, in Fig. 5 for instance, and the very curious, and as yet unexplained, Alkins effect. There may be no correlation whatever between them, but the general shape of the curve does fit in with the belief that there may be some inherent connection.

The authors' computed curves fit on the experimental ones with really remarkable agreement up to round about 40%. That is a matter of interest in two connections. First of all, it further justifies the technique which they have adopted, and, if one may say so, it also throws a great deal of light on the accuracy of the measurements for which Dr. Ford was responsible. Above about 40%, however, the discrepancy begins to increase comparatively rapidly.

Is it possible that an explanation may be found on these lines? The authors say that they have used a constant value of the factor β . As Dr. Ford pointed out at the beginning of his contribution to the discussion, there is a general trend for β to decrease as the number of passes and the reduction increase. Referring to the formula at the foot of p. 67, for a given value of the torque, if too high a value of β is taken, then the value of the calculated load will be correspondingly low. If instead of a constant value of β , a slowly changing one is employed, there is an even better agreement between the computed results and the experimental values than the curves at present would suggest.

In Fig. 1 the resultant force P is shown acting perpendicularly to the surface of the strip. Is this necessarily the case? If one were concerned solely with the weight of the roll, that must necessarily act vertically, and the resultant must be vertical; but I am not satisfied in the case of a roll in a housing that that would necessarily be so. If, instead of being perpendicular to the surface of the strip, the angle of this resultant tilts slightly, it will inevitably affect the value of β , and presumably in the direction of making it smaller than it was originally. We may have here at any rate the possibility of an explanation of the general trend which the figures given in Tables II, III, and IV show.

I now propose to be quite heretical. The authors say that they have used "cold rolling". I want it to go on record that, in my opinion, there is only one sort of rolling, and that is always cold rolling. The apparent difference between "cold" and "hot" rolling has nothing to do with the rolling process itself, but is something which happens after the rolling is completed and the pressure of the rolls has been relaxed.

DR. MAURICE COOK (*in reply*): Regarding Dr. Ford's remarks about those formulæ already published, I would point out that in developing the present method of computing rolling loads we have not been primarily concerned with the relative merits of these other methods, but it would be quite incorrect for

* Professor of Metallurgy, The University, Manchester.

any one to assume that we were of the opinion that they were without use or value. What we do maintain, however, is that the results computed by any of these methods are likely to be at considerable variance with actual values, since in using these procedures assumptions have to be made regarding frictional conditions in the roll throat, roll deformation is ignored, and, finally, it is assumed that the deformation of the material being rolled is homogeneous in character. The extent to which these results are affected by the assumption that the deformation is homogeneous is, perhaps, not very great, and I am interested to hear that Dr. Ford has some definite evidence regarding this matter. Where the limited amount of actual rolling data which the method requires for its use is not obtainable, it may well be, until more progress in the direction of a complete solution of the dynamics of rolling has been made, that, as Dr. Ford has suggested, a judicious combination of the method presented in this paper with those now available is likely to provide the most suitable means of obtaining the desired information.

With regard to the constancy of the factor β (the ratio of the lever arm to the horizontally projected length of the arc of contact), it is very satisfactory to hear that Dr. Ford has found, when roll flattening is taken into account, that the validity of this assumption is confirmed.

In answer to Mr. Hesseberg, we, in the course of our work, did not take cognizance of the possible effect of speed of rolling, but there is no reason for supposing that it would be in any way pronounced. There are other factors as well as rolling speed which we have not considered, such as the application of tension either on the entry or the exit side of the rolls. We are very interested indeed to see that Mr. Hesseberg has used our method to derive a means of obtaining rolling schedules in which a constant load is involved, for it was to facilitate exactly that sort of application that we concerned ourselves with establishing a more direct and practical means of computing rolling-mill loads.

Professor Thompson raised several interesting points, but since we have not been concerned in the work with metallurgical features, we have not given any consideration to the possible significance, or metallurgical interpretation, of the shape of the curves.

The AUTHORS (*in further, written, reply*): The meaning of our remark on kinetic energy quoted by Mr. Bland seems to have been incorrectly interpreted, for the point we intended to make was that since forward slip is only of the order of 1%, no appreciable error would be introduced if it were assumed that the exit speed of the strip was the same as that at the neutral plane, the increase in kinetic energy between this plane and the plane of exit being therefore negligible. We quite agree, of course, that this assumption could not be made if front tension were used during rolling, and we made it abundantly clear that the assumption was made only in respect of conditions not involving tension. By suitable modifications, however, the method could be employed to cover rolling conditions involving tension. While we have no proof that the lack of constancy in the value of β is responsible for the scattering of the points on the total-energy curve, further evidence may show this to be so.

Professor Thompson's suggestion of a refinement by using slowly changing values of β is interesting, and one of which we have not been unmindful, since it may be of significance in other conditions of rolling than those considered in the paper. We agree that the resultant force P does not necessarily act perpendicularly, but for the conditions investigated, the angle of the arc of contact never exceeded 7° and, therefore, could not significantly influence the values of calculated rolling loads so far as practical requirements are concerned, but whether it has any bearing on the trend of the values referred to has not yet been ascertained.

DISCUSSION ON PAPER BY DR. L. NORTH-COTT, MR. D. McLEAN, AND DR. O. R. J. LEE: "THE EFFECT OF SINGLE- AND MULTI-HOLE DIE EXTRUSION ON THE PROPERTIES OF EXTRUDED ALUMINIUM ALLOY BAR." *

(*J. Inst. Metals*, this volume, p. 81.)

DR. R. GENDERS,† M.B.E. (Member): Papers on extrusion are always to be welcomed. There are many possible new lines of development which have not yet been explored. I read this paper, therefore, with great interest, but found it from some aspects disappointing.

In the first place, the authors have not reviewed the existing state of knowledge as a basis for their additional work. Papers on the subject have been published from time to time, one of which ‡ refers to the extrusion of brass rod from a multiple die and deals with very much the same type of defect as the authors have described in the present paper.

Turning to the paper itself, the authors have gone to perhaps excessive lengths in making drift tests on components, drift tests on machined tubes, a variety of ring tests, and even tests on straightened-out rings, which establish that the radial defect in the bar was a source of circumferential weakness, without, however, providing quantitative comparisons relative to the stages of extrusion or other variables.

The main point of the paper, however, is the conclusions, which appear to be in opposition to some extent to established knowledge of the subject, and to take us back to the original erroneous view that the characteristic types of defect which can occur in an extruded bar are forms of "piping". It is well known that the type of flow which occurs in direct extrusion involves the cylindrical surface of the billet, which tends to turn inwards to follow a funnel-shaped path towards the die. At some stage it can enter the die if not prevented by known precautionary methods. In multiple-die extrusion the funnel formation divides to follow the flow streams into the respective apertures of the die, wherever they are. Eventually in the bar the flow structure forms a U-shaped pattern which corresponds with the dividing up of the funnel-shaped flow region. It is the boundaries of these U-shaped regions which contain any foreign material coming from the outside of the billet. In the 1923 paper ‡ it was shown that the U-shaped regions in three-hole extruded brass bar were outlined by dezincified metal, obviously from the outside of the billet. Where the ends of the U outcrop on the surface of the rod, any foreign material present can form a radial defect. On the basis of this type of observation the mode of flow for any combination of die apertures can easily be predicted.

The attempt of the authors to concentrate the funnel-shaped flow into a

* Discussion at the Annual General Meeting, London, 17 March 1948.

† Metallurgical Consultant, Chislehurst, Kent.

‡ R. Genders, *J. Inst. Metals*, 1923, 29, 279.

central die aperture has given interesting results, quite in accordance with the above-quoted conception of the mode of flow. It would be predicted that the effect of a central hole would depend largely on the spacing of the other holes and on the size of the central hole. It would seem doubtful whether there is any simple way of overcoming the possibility of surface defects by such methods, and reliance would still have to be placed on other precautionary methods, even if a central die-hole were provided.

Although the authors submit clear evidence in accord with the accepted mechanism of flow, they have put forward a different explanation, based on a central segregate. This is not supported by any examination of the flow structure of the billet, and even the billet discard is not described, although this should be the essential link of evidence in any speculation on extrusion flow. The authors do not criticize the generally accepted explanation which they propose to supplant by their own version.

The paper contains terms such as "radial flow", "particle constituents", and "segregate of intermetallic compounds" which are difficult to understand without fuller explanation. The nature of the central segregate which is assumed to occur in the billet is also not clear. In the absence of evidence it would surely be as reasonable to assume the presence of inverse segregation or surface exudation, which is not unknown in light alloys, and which would suggest that the billet surface might be involved.

The plasticine experiments (never very reliable because of low frictional effects) also carry the same questionable assumption of the axial segregate, and cannot constitute any support for the theory advanced by the authors. It would have been more interesting to use a billet consisting of circumferential layers of different colours. The results published by Greaves in 1921 might have been consulted in this connection. Coloured wax was used in that work to illustrate the flow through single- and two-hole dies.

I regret to say, therefore, that in my opinion this paper, although representing much careful work, possibly justified at the time when it was done, cannot be regarded as an established contribution to the general subject of extrusion flow. The useful evidence is in Plates XI and XII (which appear to confirm the type of flow known to occur) and in the observation that it is in the later stages of extrusion that the U-shaped flow pattern may contain zones of weakness. There appears to be no certain evidence that a central segregate existed in the billet and that the local circumferential weakness in the bar was not a consequence of the known mode of flow in the process.

MR. H. TAYLOR *: On the point which Dr. Genders raised with regard to flow in the extrusion process, I am sure that the authors are nearer to the fact, but I think they have missed points in their investigation which require emphasis in this discussion.

Fig. A illustrates a single-hole extrusion. The flow is straightforward, the metal flowing on the lines of easy flow, which leaves *D* as dead metal. This remains stationary although it is reduced in height owing to metal being dragged in on the line of shear. The outside of the billet, subject to outward

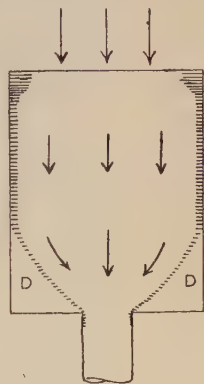


FIG. A.

* Metallurgist, Northern Aluminium Co., Ltd., Banbury, Oxon.

pressure, remains in contact with the container wall and is collected by the ram face.*

The flow through a two-hole die is very much the same except that the stream divides before the die is reached,* and the transverse flow, which in single-hole die extrusion is concentric, is not so with a two-hole die. Fig. B illustrates the two-hole die as shown in Fig. 13 of the paper (Plate XIII). Each half of the billet can be considered as a single-hole extrusion, and the transverse flow adjusts itself after this manner. Metal flows from *A* to the approximate position *B*. This causes compression in the metal flowing to the die between points *B* and *B*₁, and results in a plate-like structure. Therefore two-hole die extrusions have the general formation shown in Fig. C, *P*, termed radial flow in the paper, being plate-like in formation but otherwise similar to the fibrous structure *Q*; *R* is the highly worked metal.

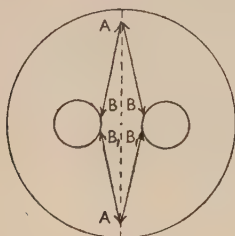


FIG. B.

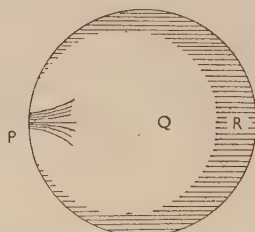


FIG. C.

The authors are correct in stating that in single-hole extrusion any segregate which exists in the centre of the billet will find its way into the centre of the bar, and, in the case of two-hole die extrusion, to the points on the peripheries of the two bars nearest to the centre of the die plate.

From that I would pass to the circumferential tensile test carried out by the authors. They show that the ultimate tensile strength is something under 1 ton/in.² less across the radial flow compared with the remainder of the periphery of the bar. In a circumferential test-piece containing the radial flow there is, therefore, a band, or area, across the middle of the test-piece, of lower strength than the remainder. The proportion of this weaker area, in any measured gauge length, will have great influence on the elongation obtained on the gauge length. If the band is narrow, the elongation over the gauge length will be low; if the band is wide, or if there are two bands (as there are in four-hole die extrusions), then the elongation will be proportionately higher. This is generally true even though the weaker area may have a higher elongation than the remainder of the test-piece. Reduction-in-area measurements would have been useful here as they would be largely independent of the size of this band.

With drift tests we have precisely the same effect. The elongation given by the weakest point governs the depth of penetration of the cone and load to fracture.

I cannot analyse the ring test very well, but it seems to me to be more of a bend test, and a bend test which is extremely harsh. It should be mentioned that the loads to fracture in Table IV, &c., in the paper, when compared for the two alloys, are not in the same ratio as the ultimate strengths of the two alloys, but fall more into line with the proof-strength ratios. If the pins for this test are made semi-circular so that they just fit snugly into

* H. Walbert, *Aluminium*, 1938, 20, 379.

the rings, then the results obtained are comparable with the circumferential tensile test.

The paper has shown that a slightly weaker area exists on the radial flow. What causes this weakness? If segregate is present to any extent there is bound to be a weakness. The authors, however, say that the material in the case of the four-hole die (i.e. three-hole with one hole in the middle) had very little intermetallic segregate, yet in the tensile test it was shown that the radial flow was weaker than the remaining periphery by approximately the same amount as before. We must, therefore, look elsewhere for the reason.

Is inverse segregation the cause? It might be expected that the alloying elements would be 10% lower at the centre than at the outside of the billet, but if this were the cause, then the four-hole die material would be weakest between the two points of radial flow.

It seems that we are left only with the structure of the material, and it should be mentioned here that the strength across the radial flow is comparable with that of a test-piece taken diametrically from the bar. A further point is that a cold reduction in excess of 25%, when carried out on the bar before heat-treatment, will modify the structure, and the resultant bar will not normally show areas of weakness.

All the material examined by the authors was free from large grain on the circumference. Had large grain been present, then the radial-flow areas would have been of greater tensile strength than the remainder of the circumference.

Summing up the points made, there is agreement with the authors that there is a small area, or areas, in bars extruded from multi-hole dies, which exhibit a slightly lower strength than the remainder of the circumference. There cannot, however, be agreement with the authors, without further evidence being offered, on the subject of ductility. With regard to the suggested method of disposing of segregate centrally situated in billets, there is agreement with its practicability, but if segregate is present in a billet, surely the ideal place to get rid of it is in the casting shop, not in the extrusion process.

DR. L. NORTHCOTT (*in reply*): I should like to say a word for the benefit of Dr. Genders. He raised two main points. One was that we did not study the previous work on the subject, which, of course, is entirely wrong; I do not know how he came to that conclusion. The fact that we did not prepare a résumé of previous work on extrusion and present it with the paper cannot be taken to mean that we did not go into the question. We are pressed repeatedly to keep our papers short, and that is the sole reason why previous work on the subject was not included.

One point which Dr. Genders did not mention is that the query which led to this work came from his own department during the war. We did not want to go into this, but it was put to us as a job which had to be done, and done quickly. The inspection side were getting very worried; they were turning down tens of thousands of these components a week, although the Services wanted them. We were asked by Dr. Genders's department to get to work on this problem.

Largely to save time, we approached industry on the subject. We realized, with our natural modesty, that we were not experts on extruded bars. Industry collaborated very well; they received us into their works and carried out any work which we asked them to do; but I think that they appreciated as much as we did that this was not an ordinary type of extrusion defect. I have had the pleasure of going through Dr. Genders's earlier paper on the subject of extrusion defects in multi-hole dies; the old type of extrusion defect, with the material from the outside of the billet turning inwards, was

certainly not responsible for this trouble. That was thoroughly appreciated by the industry.

MR. D. McLEAN (*in reply*): I think that Dr. Genders is confusing his own results with ours. Apparently he was concerned with surface defects of oxide strings which get into the bar, but that was not our trouble. The inclusions which came to the surface and caused the trouble on our bars were intermetallic particles. In Fig. 5 (Plate XI) we show at low magnification two strings of these, and if the paper and photographic position of the Institute had been better, we should have presented a number of other photographs which would have made it clear that the markings which are shown there are intermetallic particles and not oxide.

Secondly, I think that Dr. Genders's idea of where the segregate lies in the bar is not quite correct. He considered that the segregate lies along the flow structure, cutting the surface at the points marked by arrows in our Figs. 7-9 (Plate XII). Actually, however, in the three-hole and four-hole bars it lies along a chord between these positions. This is an additional reason for differentiating between the flow structure and the segregate.

DR. GENDERS: What do the arrows mean on Fig. 10 (Plate XII)?

MR. McLEAN: The arrows point to the ends of the flow structure, the points where it cuts the surface.

I think that in addition the composite billets help to show that what we are concerned with here is the central segregate of the billet, because in Plate XIII, Figs. 12-15, there is a central segregate in our plasticine composites which finishes up at these particular points on the surface of the bars.

Another point, not mentioned in the paper, is that we did some other plasticine extrusion experiments and repeated them with the lead-bismuth composites. Instead of a central core we had alternate transverse layers of differently coloured plasticine, or lead and bismuth. During extrusion these flowed in such a way that they became virtually longitudinal in the rod. It was clear from these experiments that a central segregate would go to the centre of a single-hole rod.

In general, Mr. Taylor's ideas about the flow are similar to our own. He refers to the drift test. I doubt very much whether the width in a circumferential direction of the segregated material has much influence on the properties observed. I think that the decisive factor is the fracture strength of this material. In the drift test the cylinder has to expand, and it is clearly possible for most of the expansion to occur in the non-segregated part. If it so happens that the stress-strain curve of this material is much steeper than the stress-strain curve of the non-segregated material, which is likely to be the case, fracture will occur when the circumferential stress in the segregated material exceeds the fracture strength.

CORRESPONDENCE.

DR. R. GENDERS: While the oral discussion clarified some points which were raised, it would be useful to have some definite indication regarding the position of the "segregates" relative to the flow structure as seen in the cross-sections of the bars. I took the arrows shown in the illustrations to give the positions of the incipient surface defects, and this would be in accordance with earlier work demonstrating the significance of the U-shaped flow pattern. The interior of the U corresponded to the interior of the funnel formation in the billet and was usually sound; if it contained any axial segregate, this

might flow into the edge of the bar between the outcropping branches of the U, and the position of the external lines on the bar relative to the U was thus all-important in deciding their origin. The arrows shown in the paper would suggest that the branches of the U, and therefore normal flow, were the relevant features. This view is supported by the statement that the defects appeared in the later stages of extrusion, since axial flow in the billet is relatively rapid, and would extrude any axial segregate fairly early in the process, leaving the later portions sound. Very near the end, when the small "dead" cone at the rear end of the billet passes the die, a further small portion of central segregate may conceivably reappear if a segregate extended to the rear surface of the original billet. The sequence of these various effects can be followed clearly by reference to the changes in shape and size of the U-shaped flow pattern which occur progressively during the extrusion of the billet. The subject is of practical interest, and it would be useful if any subsequent work could include such observations. It is realized, of course, that the circumstances of the work described in the paper would probably not permit the examination to be comprehensive, and that the conclusions submitted are therefore tentative. It is agreed that a written review of previous work is not important, but failing this a short list of references might be useful.

The AUTHORS (*in further, written, reply to the discussion and in reply to correspondence*): We listened to Dr. Genders's contribution to the discussion and read the printed version with some amazement. The senior author in particular was fully aware of the earlier work to which Dr. Genders refers, carried out in the same laboratories a quarter of a century ago. It is a pity, however, that Dr. Genders shuts his eyes to the possibility of there being a second extrusion defect, and his professed inability to understand the term "segregate of intermetallic compounds" floors us completely.

As Dr. Genders points out, if the segregate found in our bars were a surface defect of the original billet, it should be along the U-shaped flow pattern at the positions marked by the arrows in Figs. 8 and 9 (Plate XII), but if it were a central segregate of the original billet, it should be at the edge of the bar between these positions, along an arc or chord on a transverse section. In actual fact the segregate was found in the latter position, and this is illustrated by the plasticine models in Figs. 14 and 15 (Plate XIII). It therefore seems we can all agree that it formed a central segregate of the original billet.

In this connection, perhaps Fig. 5 (Plate XI) has caused some confusion, because without an additional photograph of the same area at a higher power it is easy to believe that the black streaks shown are oxide strings, and therefore came from the surface of the billet. However, a moment's examination at a higher power would convince anyone that they are actually strings of intermetallic particles, which therefore in all probability came from the centre of the billet.

Since attention has been focused rather sharply on the segregate in these bars, it is only fair to both manufacturers to point out that the segregate concentrates found correspond to quite a low concentration in the billet and are representative of a high-grade product.

We are in general agreement with Mr. Taylor on the question of flow during extrusion, but we are not quite clear about his argument on the drift test or circumferential tensile test. In our view, fracture will occur when work-hardening of the non-segregated material raises the circumferential stress above the fracture strength of the segregated material, causing early fracture. Because of this, and even more because so many of our measurements show multi-hole material to be less ductile than single-hole metal, we still feel that our conclusion on this point is correct.

DISCUSSION ON MR. D. McLEAN'S PAPER: "STRIATIONS: METALLOGRAPHIC EVIDENCE OF SLIP." *

(*J. Inst. Metals*, this volume, p. 95.)

MR. R. EBORALL,† B.A. (Member): In dealing with these striations the author has directed attention to some interesting effects, and his main conclusions have been well borne out by the more recent and slightly different paper by Burke and Barrett.‡ I do not want to discuss the question in a very general way, but to refer to some particular points.

The author finds that the number of striations is smaller than the number of slip lines on the surface; also that at deformations for which the slip lines extend right across the surface of a crystal the striations occur only over a limited range, generally starting at the grain boundaries, and that with higher degrees of cold work the striations extend right across the crystal. I suppose that these observations are in agreement with the normal conception of the slip process, in which the slipped area, which has an undistorted structure, is bounded by dislocations along lines roughly normal to the slip direction. Thus, if the grain boundary represents a constraint on the free movement of the crystal, each unit of slip should result in a pair of dislocations becoming stuck near the grain boundary, one on each side of the crystal. There would thus be an accumulation of dislocations along the slip plane near the grain boundary, and this would constitute the real disturbance to the structure which gives rise to the etching effect. I feel that the lumpy structure shown in Fig. 7 (Plate XVI), and the author's reference to mosaic blocks, are really irrelevant, and that this structure is primarily due to a random roughening action of the particular etchant used.

The first occurrence of striations at the grain boundaries ties up nicely with the fact that, on subsequent annealing, the recrystallization nuclei, in some cases at any rate, form preferentially at the grain boundaries if the deformation has been small, and appear within the grains after higher deformations.§ Incidentally, Burke and Barrett show in their paper a structure in which there are recrystallization nuclei associated with striations.

The AUTHOR (*in reply*): I think that the occurrence of striations first at the grain boundaries is quite understandable, whether one works on the dislocation hypothesis or not, because at the grain boundaries the lattice must be irregular and the adjoining crystal will prevent the regular structure being maintained after the deformation.

With regard to Fig. 7, I quite agree that what one sees is the surface roughness of the crystal, because that is what the electron microscope shows up; but there surely must be something in the metal to cause that surface roughness. With copper, other electron-microscope studies have shown the same sort of structure, and, since the size of the blocks is about that generally accepted for the so-called mosaic blocks, it seems possible that this is the mosaic structure. These matters, however, are somewhat nebulous at the moment.

* Discussion at the Annual General Meeting, London, 16 March 1948.

† Head of General Metallurgy Section, British Non-Ferrous Metals Research Association, London.

‡ J. E. Burke and C. S. Barrett, *Metals Technol.*, 1948, 15, (2); *A.I.M.M.E. Tech. Publ. No. 2327*.

§ P. Lacombe and L. Beaujard, *Rev. Mét.*, 1947, 44, 71.

DISCUSSION ON PAPER BY DR. P. BRENNER AND DR. W. ROTH: "RECENT DEVELOP- MENTS IN CORROSION-RESISTANT ALU- MINIUM-MAGNESIUM ALLOYS." *

(*J. Inst. Metals*, this volume, p. 159.)

DR. F. A. CHAMPION † (Member): The authors have presented data from comprehensive experiments over a wide field of the variables which may affect the corrosion-resistance of aluminium-magnesium alloys. I feel that it is unfortunate, therefore, that the methods of corrosion testing which they have employed make one hesitate to use the results of those tests as extensively as would have been possible with more reliable testing methods. The majority of the tests, as Dr. Brenner has emphasized, have been carried out under stress-corrosion conditions. A few tests have been made in the absence of stress, but the corrosion conditions are not comparable, and therefore we can get no information from these results as to whether the alloys are susceptible to stress-corrosion; that is, whether corrosion has been accelerated by stress. Moreover, in most cases the criterion of corrosion-resistance has been the time taken for the strength to decrease to a definite value of either 10 or 15 kg./mm.² instead of to a percentage of the initial ultimate strength or proof stress, as is more usual. The former method, I feel, gives more favourable results for alloys of higher initial properties, since a greater margin for loss is allowed.

In deciding on the corrosion conditions to be adopted for most of the tests, the authors appear to have paid more attention to rapidity of corrosion and to convenience than to representation of service conditions. Thus, the solution mainly used contained 3% sodium chloride and 1% hydrochloric acid. I believe that this was originally developed in this country ‡ as a rapid, more or less qualitative means of exploring the tendency for some aluminium alloys to show intercrystalline corrosion. This solution has a pH of 0.9, and from my own experience I feel that even for qualitative observations such an acid solution must be used with caution, while quantitative observations with this solution are unreliable. The authors give results in Table X which they regard as justifying their accelerated tests, but I should like to take some (mean) values from their Table X which are of interest in this connection:

	Sea Water.	Lab. Test 1 (NaCl).	Lab. Test 3 (NaCl + HCl).
7% Mg, quenched . . .	8	5	3.3
9% Mg, quenched . . .	2.3	2.3	1.3
9% Mg, furnace cooled . . .	>140	>140	3.7

These are mean values for aged metal taken from that table, and the figures do not seem to me to be very encouraging or to give one much faith in that solution.

* Discussion at the Annual General Meeting, London, 18 March 1948.

† Research Laboratories of the British Aluminium Co., Ltd., Chalfont Park, Bucks.

‡ A. J. Sidery, K. G. Lewis, and H. Sutton, *J. Inst. Metals*, 1932, **48**, 165.

Passing now to the authors' discussion of the reasons for the results obtained, they correlate the corrosion-resistance and the structure of the alloys, but I am somewhat concerned about that correlation, because if you take the results in Tables VI and VII and compare them with the corresponding micrographs in Plates XIX and XX, the results appear to contradict the theory which is put forward by the authors. Thus, Figs. 12 and 19 show "a discontinuous network", to use the authors' words; the corrosion tests gave lives under stress-corrosion conditions of 7 days (Fig. 12) and 3.5 days (Fig. 19). Figs. 15 and 17 are for a similar alloy with different ageing treatments giving a more continuous network, and for those we get lives of 12 days (Fig. 15) and 17.5 days (Fig. 17). In both cases with the more continuous network we have a longer life, which is the converse of what is suggested in the paper. These discrepancies, however, may be due to the unreliability of the particular accelerated corrosion test which has been used; but I note that the authors have used that test for the routine control of commercial production, indicating that they feel that it is more reliable than micro-examination of the structure. I should much appreciate their comments on whether the corrosion behaviour can be predicted from an examination of the structure.

The other point on which I want to touch is the effect of copper on these alloys. The results given in Table IV suggest that the addition of 0.9% copper to the 7%-magnesium alloy, while it decreases the corrosion-resistance of the homogenized alloy, increases the resistance of the alloy when aged for 3 days at 100° C. It would be interesting to know whether this increase in corrosion-resistance is also observed with other ageing conditions (time and temperature). Also, in Tables III and IV, for a number of the alloys the copper content is given as "Nil", and it would be helpful if the authors could give an interpretation of that term.

MR. G. J. METCALFE,* M.Sc.Tech. (Member): The authors have covered a very large field in this paper, and many of the points which I had intended to raise have already been covered by Dr. Champion. I think, however, that the metallographic examination and corrosion tests which the authors have carried out have established the close relationship of susceptibility to intercrystalline corrosion and stress-corrosion of the aluminium-magnesium alloys with the presence of a grain-boundary precipitate.

As far as the accelerated test is concerned, they claim that it has been a useful check on material before distribution to the user. With regard to this point, I think that we should remember that they use it only for one particular type of alloy, the aluminium-magnesium alloy, and it would be wrong to attempt to use it for other aluminium alloys.

With regard to the definition of the term "stress-corrosion", in August 1945 Dr. Sutton, Mr. Liddiard, Dr. Chalmers, and Dr. Champion wrote a letter to the Institute of Metals in which they suggested a tentative definition for the term "stress-corrosion", and I should like to read the suggestion which they made:

"The term 'stress-corrosion' implies a greater deterioration in the mechanical properties of the material through the simultaneous action of a static stress and exposure to corrosive environment than would occur by the separate but additive action of those agencies."

As we all know, Dr. Brenner has published several papers on the stress-corrosion of aluminium alloys, but it does seem that his interpretation of the meaning of this term is rather different from that suggested in the passage

* Fulmer Research Institute, Stoke Poges, Bucks.

from the letter which I have just read. I should be grateful if Dr. Brenner would enlarge upon this and give us a better idea of what he really means by that term, and also if he would tell us whether other German workers in this field hold the same views.

In their tests, the authors stressed the specimens in bending. I should like to know what advantage they claim for that, apart from simplicity of set-up, over direct tensile loading. Crack propagation, no doubt, would be more rapid in bending than in direct tension, since the deeper the crack the greater the stress at the root.

There seems to be some slight difference of opinion as to the effect of copper when we compare this work with the work of Sterner-Rainer * and Seemann and Wesch,† who suggest that the addition of even 0.05% copper causes a serious increase in the susceptibility to intercrystalline corrosion and stress-corrosion. The present authors claim that 0.9% copper improves the properties in the aged condition. In view of the growing popularity of these alloys, I think that this field should be more thoroughly investigated.

The authors used a reduction of 20% in their investigations of the effects of cold rolling followed by ageing. Have they any particular reason for choosing this figure? In some work on an aluminium-5% magnesium alloy, I found that increasing the amount of cold rolling up to 20-30% reduction in thickness gradually increased the amount of grain-boundary precipitate formed when the material was aged afterwards at 70° C. With higher reductions, precipitation began to take place in the grains as well. It seems to me that instead of slow furnace-cooling, the material could be heavily cold rolled (60-70% reduction in thickness) and then aged, producing a state of susceptibility to general corrosion attack, but a decrease, or possibly an elimination, of susceptibility to intercrystalline corrosion or stress-corrosion.

If susceptibility to intercrystalline attack is reduced by this treatment, then the resistance to intercrystalline attack of the higher magnesium alloys may be reduced by shot-peening the material before ageing.

In the discussion of their results, the authors raise the question of the effects of internal stresses produced by different cooling methods, the distribution of these stresses, and their influence on precipitation. In addition to the effects of internal stress, might not micro-segregation play some part? Have the authors any information on the effects of micro-segregation on the distribution of the precipitate in these alloys?

DR. A. G. QUARRELL ‡ (Member): I have read this paper with great interest, but, as other speakers have emphasized, there is a great difference between the points of view regarding stress-corrosion adopted in Germany and in this country, and, unless this is clear, many erroneous conclusions may be drawn about the susceptibility of the aluminium-magnesium alloys to stress-corrosion. I think that it is fairly clear that when the authors have reported failure by yielding, this failure is probably caused by general corrosion of the specimen. This is not surprising, since rapid and continuous evolution of hydrogen occurs when aluminium-magnesium alloys are immersed in a solution containing 3% sodium chloride and 1% hydrochloric acid.

It is not so clear that failure by fracture is caused by what we should call stress-corrosion, although at first sight this seemed likely. In Table IV, however, the authors report failure by fracture of a homogenized 7%-magnesium alloy containing copper. It is interesting to note that the

* R. Sterner-Rainer, *Korrosion u. Metallschutz*, 1940, **16**, 278.

† H. J. Seeman and K. Wesch, *Korrosion u. Metallschutz*, 1940, **16**, 256.

‡ Research Manager, British Non-Ferrous Metals Research Association, London.

duplicate specimen failed by yielding in approximately the same time, but I should particularly like to know whether the fractured specimen was really homogenized and free from precipitate, and whether the authors think that this specimen failed by what we should call stress-corrosion rather than by stress-corrosion on the German definition. If this is so, it is of considerable importance, because it would make it necessary for us to revise our ideas as to what stress-corrosion really is. Experience in this country would, I think, confirm the view put forward by the authors, that homogenized aluminium-7% magnesium alloy is quite immune to stress-corrosion, and one would not expect the presence of copper to affect this in the absence of precipitation.

Like Dr. Champion, I found a great deal to interest me in Table X; not only is there rather poorer agreement between the results of the tests in the acid sodium chloride solution and exposure to sea water than there is in the case of the neutral solution, but I am not clear what advantage is gained by using the acid solution. For example, if we look at the times to failure of what we might call the susceptible specimens, in most cases they are not significantly shorter in the acid solution than in the neutral solution. In the case of relatively unsusceptible materials, such as gave lives greater than 140 days in the North Sea, there is very much better agreement between the 3% sodium chloride solution and the sea-water tests. With these specimens, the acid solution gives shorter lives and appears to differentiate between different specimens, although as far as I can see, the authors cannot say whether that differentiation is a real one or not as the tests in the North Sea have not gone on for sufficiently long periods. I consider that the authors have not made the point that this solution gives a good guide to probable service behaviour.

Reference has already been made to the fact that the authors suggest that extreme susceptibility results from the presence of a more or less continuous grain-boundary network of precipitate. This certainly does appear to be true at first sight when working with these aluminium-magnesium alloys, but we have recently observed structures where the precipitate is in quite clearly defined particles at the grain boundaries, and where the material has been quite susceptible to stress-corrosion. I am afraid that we cannot give the full explanation of it at this stage, because we do not know it yet.

In Fig. 27, p. 187, the authors compare the behaviour in corrosion of a continuous network and isolated particles of precipitate respectively. It should be emphasized, I think, that the conclusions which they draw relate only to the case where the precipitate is anodic to the main body of the crystal. If, on the other hand, the precipitate is cathodic, then the distribution of isolated particles as shown in Fig. 27 (b) (p. 187) can very well, I think, lead to continuous penetration of the metal, the attack initiated by one cathode passing on to the next. I think that sometimes we do not realize this sufficiently in discussing stress-corrosion.

I am sorry that most of my criticism has been rather destructive, but it is directed against the definition of stress-corrosion adopted in Germany, and not against the work which the authors have carried out. The paper is probably the most comprehensive treatment of the behaviour of aluminium-magnesium alloys under corrosion conditions which has yet been published; would it be too much to ask the authors to give a brief summary of their work interpreted in the light of our own definition of stress-corrosion?

MR. C. EDELEANU,* B.A. (Student Member): I agree that the tests carried out in acid solution may not be entirely satisfactory to those interested in service behaviour, but in the definition of stress-corrosion the electrolyte is not

* Cambridge University.

specified. As long as corrosion is accelerated by stress we are dealing with stress-corrosion, and it is immaterial whether the solution is lemon juice or sea water.

My second point deals with the microscopic examination. Etching is a corrosion experiment which is not essentially different from corrosion by sea water. If in a given condition the material shows continuous black lines at the grain boundaries, we can assume that a continuous trench has been formed during etching. It is not surprising that such a specimen when exposed to prolonged corrosion by sea water will fail ultimately in an intercrystalline manner. What is not established, I feel, is the nature of the dissolved material. It is generally assumed that it consists of the β phase, and although that may be correct I would like to mention that Lacombe and Beaujard* have described the intercrystalline corrosion of very pure aluminium caused by purely physical reasons. I would also like to mention an effect which has possibly been noticed by other workers. On etching a specimen containing large particles I have noticed a white central spot surrounded by a black line. Corrosion seems to be taking place at the interface between the α solid solution and the β phase, and not on the particle itself. The effect may, of course, be due to current-density effects.

My last point deals with strain. Many workers have shown that strain accelerates precipitation. On quenching, internal stresses are introduced in the material, and these can be responsible for the greater susceptibility obtained after ageing. The authors mention in the last part of their paper that quenching stresses on the surface would be compressive, and they claim that these forces may be of some value in lengthening the life of specimens. I do not think that is so. When a specimen in this condition is exposed to temperatures sufficiently high to cause precipitation it will become more susceptible than a specimen properly annealed. Some of my own experiments have shown that if specimens are scratched just before ageing, their life is shortened by about one half. If they are scratched at any other time, before solution treatment or after ageing has been completed, their life is hardly affected. In the first case failure always occurs at the scratch and in the other cases it does not, so that it is not a notch effect. It is an effect due to precipitation at a place where there is strain. I feel that the compressive strain introduced on quenching is therefore rather dangerous.

DR. ULICK R. EVANS,† M.A. (Member): The authors state that "penetration may be accelerated by differences in aeration", and that "the current density, and therefore the corrosion rate, is higher, the smaller the anodic surface in proportion to the cathodic surface". Both those statements are probably correct, at least under the conditions commonly met with in service, but it is important to note that the argument advanced will be valid only if the control is mainly *cathodic*. If the control was solely *anodic*, then, when the anodic area was decreased, the corrosion rate and current flowing would be decreased proportionately; the current density would remain, therefore, unchanged, and the intensity of corrosion would not be increased.

Similarly, if the resistance of the path joining the anodic and the cathodic areas were appreciably to influence the current flow, the conditions would become less favourable for the uptake of oxygen at an external cathode, with corrosion occurring at distant anodes (the tips of the cracks). Clearly, the shape of the cracks may be important. At one time it seemed difficult to picture current flowing between the external cathode and the tip of an advancing crack, because the crack was regarded as a V-shaped notch, which

* *J. Inst. Metals*, this vol., p. 1.

† Cambridge University.

would impose a high electrolytic resistance; Edeleanu * has recently shown that the outer portions of the cracks may in some circumstances be comparatively broad, with almost parallel walls; there may be narrower channels penetrating further into the metal, but at any rate the electrolytic resistance of the circuit must be smaller than was at one time imagined, and the mechanism suggested by the authors appears to be justified.

It is stated that the beneficial effect of the internal stresses should not be under-rated; this is readily admitted, but, on the other hand, it should not be over-rated. Actually, internal stresses will only be beneficial if they are compressional, and thus capable of counteracting imposed tensional stresses; even in that case there may be unfavourable secondary effects, as Mr. Edeleanu has pointed out. In the case where there is a random distribution of tensional and compressional stresses, the effect of the stresses is likely to be wholly unfavourable. At places where the internal stresses operating are tensional, they will certainly help the applied tensional stresses, and will favour stress-corrosion cracking; at other places, where they are compressional, they will then oppose the applied tensional stresses, and prevent cracks from developing at points where otherwise they would occur. Thus those cracks which do develop will be spaced further apart as the result of the internal stresses. Now, it is well established that stress intensification due to well separated cracks is much more serious than that due to cracks which occur close together. It may, therefore, be expected that both the propagation of the cracks at certain places and the absence of cracking at the intermediate places are both unfavourable factors. It appears necessary, therefore, before claiming that internal stresses are favourable, to obtain some idea of the orientation and also of the distribution of the internal stresses which are operating.

MR. A. J. MURPHY,† M.Sc. (Vice-President): I am very concerned about the encouragement which this paper gives to the idea that a continuous film of precipitate in the grain boundaries is a requirement for short life under stress-corrosion in aluminium-magnesium alloys. I do not think that that is correct. Dr. Quarrell mentioned a case of severe reduction of life under stress-corrosion when microscopical evidence did not disclose a continuous film at the grain boundary, and I would say that in view of the importance attached to this point, the microstructural evidence which the authors produce is not convincing. In Fig. 16 (Plate XX), as far as I can see, there is no resemblance of a continuous film in the grain boundary, and in Fig. 21 (Plate XX) it is not possible to say whether there is or is not a large proportion of continuous intercrystalline precipitate.

The explanation advanced does not appear to depend on the material being in the wrought state, and therefore evidence from cast material ought to be admissible. In the case of the 10%-magnesium alloy in the cast state, it is not at all easy to produce failure under stress-corrosion, but the situation as regards what is the most damaging treatment is exactly the reverse of that of which the authors have spoken in connection with the 7%-magnesium wrought alloy. In an investigation with the 10%-magnesium alloy, after a solution treatment at 430° C., two alternative treatments were applied: one sample was quenched into boiling water and the other was cooled very slowly in the furnace. The proof stress was 11 tons/in.² in the first case and about 8 tons/in.² in the second. When each specimen was loaded to its proof stress and subjected to spray by sea water, the material which had been slowly cooled fractured in 12 hr., whereas that which had been quenched into boiling

* Quoted by U. R. Evans, *Symposium on Internal Stresses in Metals and Alloys* (Institute of Metals), 1947, p. 295, Figs. 15, 16.

† Director and Chief Metallurgist, J. Stone and Company, Ltd., London.

water survived for over 700 hr. On examining the microstructure, the characteristic of the material quenched into boiling water was a relatively small amount of precipitate, certainly not continuous, in the grain boundary, whereas the other material had a large amount of precipitate which was, however, still in isolated particles in the grain boundary. In view of these results it is undesirable that the impression should be given that the absence of a continuous film in the grain boundary is a reliable criterion for satisfactory behaviour under stress-corrosion conditions.

MR. E. C. W. PERRYMAN,* B.A. (Student Member): During work on the stress-corrosion properties of aluminium-magnesium alloys at the British Non-Ferrous Metals Research Association we have found that when using an acid solution, such as that used by the authors for their stress-corrosion tests, a large amount of general corrosion takes place in addition to stress-corrosion. It is a pity, therefore, that the authors did not carry out corrosion tests on unstressed specimens alongside their stress-corrosion tests, so that the strength lost owing to stress-corrosion and that lost owing to general corrosion could have been separated. The authors have used throughout their paper time to failure as a criterion of the stress-corrosion susceptibility. This can sometimes lead to false conclusions, for if we imagine two materials of widely different tensile strengths which would be attacked at the same rate, the stronger material under the same conditions of stress and corrosion would give the longer life, because the crack has to advance further before the cross-sectional area is reduced sufficiently for the tensile strength of the material to be exceeded.

With regard to those homogeneous specimens which the authors describe as having failed by yielding, I should like to ask them the following question. If the same specimen had been corroded for an equal time unstressed, and then tested for mechanical properties, would the residual strength after corrosion be equal to the stress at which the specimens failed in the authors' stress-corrosion tests? It is possible that stress might increase the rate of general corrosion, in addition to producing the stress-corrosion cracking.

Turning to Table III, p. 169, one of the homogeneous alloys, No. 5, is stated to have fractured, from which I presume that it failed by stress-corrosion, with an intercrystalline fracture. In view of the authors' general conclusion that the stress-corrosion susceptibility depends on the continuity of the grain-boundary precipitate, I find it very difficult to reconcile these two facts.

On p. 165, the authors state that they could not see any precipitate after ageing the aluminium-9% magnesium alloy for 3 days at 75° C. or 4 hr. at 100° C. Was the material used of high purity or commercial purity, because they do not mention this in the paper? On p. 170 they also say that they saw no difference in the amount of precipitate in the super-pure and the impure aluminium-7% magnesium alloy containing iron, silicon, and manganese. Ageing experiments which we have carried out on homogenized commercial-purity aluminium-7% magnesium alloy containing silicon about 0.2, iron 0.3, and manganese 0.3%, have shown that after treatments such as those described by the authors, 3 days at 75° C. or 4 hr. at 100° C., the β phase is precipitated at the grain boundaries, though not forming a complete intergranular network. With this 7% magnesium alloy we have also found precipitate at the grain boundaries after much shorter times of ageing, e.g. 4 hr. at 75° C. Similar experiments on a wrought super-purity aluminium-10% magnesium alloy showed that no precipitate was produced after such short ageing treatments, although one would expect more precipitation with the higher magnesium content. One is forced, therefore, to conclude that the

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rate of precipitation depends to a large extent on the degree of purity of the alloy under consideration, and it may be that this variation in the rate of precipitation of the second phase with the purity of the alloy could explain the difference between the stress-corrosion-resistance of the high-purity alloys and that of the low-purity alloys used by the authors.

Turning to the effect of small additions of other elements to the aluminium-7% magnesium alloy, we have found that small additions of zinc decrease the rate of precipitation on ageing after solution treatment. When, however, the alloys containing zinc are heat-treated to produce a continuous network of the second phase, we have found no difference in the stress-corrosion-resistance of the alloys with and without zinc. The zinc additions thus lengthen the time of ageing at any one temperature necessary for the formation of a completely continuous network of the second phase, this structure giving a very poor stress-corrosion-resistance whether zinc is present or not.

In their discussion of results, the authors suggest that differences of aeration may play some part in the stress-corrosion susceptibility of the alloy. As the test used was one in which the specimen was partially immersed, I should like to ask them whether their failures took place at the liquid level or not, for if differential aeration was playing a part then one would expect fracture at the liquid level.

CORRESPONDENCE.

DR. G. SIEBEL and DR. H. VOSSKÜHLER *: Detailed investigations of the properties of wrought aluminium-magnesium alloys with high magnesium contents, carried out by the metallurgical research laboratories of I.G. Farben-Industrie, Bitterfeld, after the introduction of these alloys into practical use, revealed that these alloys are susceptible to stress-corrosion. Homogeneous cold-worked aluminium alloys with between 7 and 9% magnesium are especially susceptible to stress-corrosion failure after a final heat-treatment at 75°-200° C. The microstructure of these alloys so treated revealed continuous grain-boundary precipitation of the intermetallic compound Al_3Mg_2 .

Various methods were used to stabilize the alloys, resulting in the known heterogenizing processes. These processes were aided by the addition of small amounts of alloying elements, such as manganese, chromium, zinc, titanium, silicon, and calcium.

The following methods were investigated :

(1) Slow cooling from the homogeneous state (in the furnace, in air, oil quenching, and hot-water quenching).

(2) Cold-water quenching or cooling in air from the homogeneous state, followed by heterogenizing just below the solid-solubility temperature (in an air furnace or salt bath) and water quenching or cooling in air.

(3) Heat-treatment at the homogenizing temperature, then cooling to and holding at a heterogenizing temperature (in an air furnace or salt bath), followed by water quenching or air cooling.

(4) Methods (1)-(3) with intermediate cold working.

Many difficulties arose when the heterogenizing processes (1)-(3) were used on an industrial scale, owing to the fact that precipitation of the intermetallic compounds did not occur in the required coagulated form. Also the required degree of heterogeneity could not be obtained with the necessary precision.

We did not favour slow cooling of the alloys as proposed by Brenner and Roth, even though it produces alloys resistant to stress-corrosion, the reason being that with slow cooling, good mechanical properties and formability are

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not obtained with certainty, especially with alloys containing 9% magnesium, in the half-hard condition, on account of their considerable heterogeneity. Furthermore, furnace cooling does not appear to be economical.

A marked improvement in the heterogenizing process was obtained by keeping the homogenizing temperature low and working according to method (4).^{*} It was also found that the alloying additions mentioned above improved the stress-corrosion behaviour if the proper heat-treatment conditions were used.

The results of stress-corrosion tests (loop tests using an alternating-immersion apparatus with artificial sea water) on 1 mm.-thick samples of an aluminium-magnesium alloy containing approximately 9% magnesium and in the half-hard condition are given in Table A.

TABLE A.

Treatment of Samples.	Temperature, ° C.	Life of Loops, days.
72 hr. anneal	100	> 320
48 hr. anneal	150	> 320
30 hr. anneal	200	> 320
20% cold deformation + 48 hr. anneal .	100	> 135

Contrary to Brenner and Roth's observation (viz. after complete dissolution a stable state is obtained that is also resistant after low-temperature annealing) that a relationship exists between the structure of the alloy and its stress-corrosion behaviour, we have observed during our investigations that a high resistance to stress-corrosion is related to proper heterogenizing conditions with a structure of low degree of dissolution. Coagulated precipitation at the boundaries or within the grains is no criterion of high resistance to stress-corrosion, but, generally, continuous precipitation at the boundaries results in a low resistance to stress-corrosion.

The use of a 3% NaCl, 1% HCl solution as a corrosive medium does not correspond to practical conditions. During sea-water corrosion, alkaline conditions are present, which naturally do not affect the grain-boundary precipitate (Al_3Mg_2), which is resistant to alkaline solutions; but acid solutions result in intercrystalline failure. Solutions containing acid chlorine cause chemical attack on heterogeneous alloys, and therefore the results obtained cannot be compared with those obtained on homogeneous alloys. These facts show the disadvantages of corrosive media containing acid chlorine in solution. To increase the speed of the attack during tests, it seems better to produce alloys having high susceptibility to stress-corrosion rather than to increase the strength of the corrosive medium.

DR. H. SUTTON † (Member): The authors have given us a detailed description of a very comprehensive investigation embracing a lengthy series of experiments. They have clearly demonstrated the advantages to be gained by special methods of treatment which produce a structure in which the β phase is distributed generally in the form of separate particles. One point which interests me very much is the effect of small or moderate amounts of deformation following such treatments, especially when the material is initially in a condition of favourable structure (Table IX). Metcalfe ‡ found

* G. Siebel and H. Vosskübler, *Aluminium*, 1942, **24**, 129. Detailed report by G. Siebel and H. Vosskübler is given in *Metall*, 1948, (9 10), 141-146.

† Ministry of Supply.

‡ G. J. Metcalfe, *J. Inst. Metals*, 1946, **72**, 487.

that cold working, as in pre-forming the head on a rivet, had a harmful effect on the resistance to intercrystalline corrosion in material (containing 5% magnesium) having initially a reasonably good resistance to intercrystalline corrosion. I would like to ask the authors whether they think that the effect is associated with precipitation of small amounts of β or β' from solid solution containing just a little more than the equilibrium amount at the temperature concerned, precipitation being facilitated by strain.

The results of the beach-exposure tests are of particular interest. Such tests have a more satisfying nature than accelerated laboratory tests. Only by correlation studies such as those summarized in Table X of the authors' paper can a safe approach be made to the interpretation of accelerated laboratory tests.

Several investigators* have claimed that manganese additions greater than normal give improved corrosion-resistance. It would be interesting to know whether the authors have any experience of the effect of high manganese content on stress-corrosion-resistance. According to Akimow and others,† the Al-Mg-Mn constituent is anodic to the Mg_2Al_3 , which is in turn anodic to the solid solution. I would be glad to know if the authors have any evidence of that kind.

With reference to Dr. Champion's remarks about the 3% NaCl, 1% HCl (by wt.) solution, it is true that Sidery, Lewis, and I ‡ used it in our early work on intercrystalline corrosion in alloys of the Duralumin type. We described results obtained with other corroding media also. My own impressions are that the solution is a useful one when observing the tendency to intercrystalline corrosion in alloys of the Duralumin type, especially when a "short-time" test is necessary. The solution seems to me to be rather acid for long-time stress-corrosion tests at normal temperatures, but it is of interest to know that the authors found it useful in making short-time stress-corrosion tests on commercially produced sheets.

The AUTHORS (in written reply to the discussion and in reply to correspondence): We wish to thank all contributors to the discussion on our paper for their interest. Before replying to the great number of questions relating to testing methods and results described in the paper, we will try to give a definition of the term "stress-corrosion" as we understand it.

We feel that the definition mentioned by Mr. Metcalfe, as well as the American definition: "Accelerated corrosion of metal produced by externally applied or internal stress" § are restricted to tension stresses and therefore may not cover the whole field of stress-corrosion. Though few tests have been made under compression, there is some evidence that compression stresses do not accelerate corrosion and that they may even have a beneficial effect. At least in the present stage of research on stress-corrosion, we would prefer a more general definition which includes all kinds of stress and corrosion attack, as for example: "The term 'stress-corrosion' implies the effect of simultaneous static stress and corrosion".

We would not like to restrict the failures by stress-corrosion to intercrystalline or transcrystalline cracking, since stress has probably also an effect on corrosion by pitting or general attack. With the applied bending-

* L. N. Sergeev and B. I. Rimmer, *Trudy Vsesoyuz. Alyumin.-Magn. Inst.*, 1939, (19), 197, 246.

† G. Akimow, *Korrosion u. Metallschutz*, 1932, 8, 197.

V. O. Krenig and S. M. Ter-Osipyan, *Metallurg*, 1940, (9), 5.

‡ A. J. Sidery, K. G. Lewis, and H. Sutton, *J. Inst. Metals*, 1932, 48, 165.

§ A.S.T.M.-A.I.M.E. *Symposium on Stress-Corrosion Cracking*, December 1944.

corrosion test, "fractures" (produced by brittle cracking) as well as "yielding" (produced by high plastic deformation) are obtained, and both types of failure have been regarded as stress-corrosion failures in the paper, because they are produced by simultaneous stress and corrosion. Fractures are generally caused by preferred intercrystalline penetration corrosion at the place of the fracture (notch effect). Yielding may occur by general corrosion or by uniform penetration of intercrystalline corrosion all over the surface of the specimen which has a similar effect to general corrosion. The different types of attack were frequently observed in the same specimen, as for example: intercrystalline corrosion + pitting, general corrosion + intercrystalline attack, and so on.

In the discussion much valuable criticism has been made in regard to the applied accelerated stress-corrosion testing method, especially concerning the acid-containing electrolyte. It is difficult to refute the objections raised because most of them can be directed against any accelerated test. Even in the case of the usual outdoor tests the conditions do not correspond to service conditions, and the results of such tests must be applied with caution. This is true to a higher degree of accelerated tests.

On the other hand, accelerated corrosion tests make it possible to investigate a wide field of variables in a reasonable time, as shown for example in the paper. Such tests may be useful even if their results do not agree with results obtained under service conditions, especially in research where it is of interest to investigate the corrosion properties and the mechanism of corrosion under any defined conditions.

Of course, the practical importance of an accelerated test is much greater if its results correlate with results obtained under service conditions. It was very surprising that in the case of aluminium-magnesium alloys we found a fairly good agreement between the results of the accelerated stress-corrosion tests in a 3% NaCl solution containing 1% HCl, and the sea-water tests, though the conditions of chemical attack are very different. In these circumstances one cannot expect an exact agreement, but we did obtain a certain classification of the investigated materials which was practically the same in the accelerated test as in the sea-water test. This is also true for the results which Dr. Champion took from Table X, where the three selected materials show the same order in their stress-corrosion properties in the different stress-corrosion tests. Thus, in all three tests the aluminium-9% magnesium alloy, furnace cooled and aged, gave the highest stress-corrosion-resistance, and the same alloy, quenched and aged, the lowest resistance. We agree that the lack of a quantitative agreement between the results of the three testing methods is not satisfactory, but this is perhaps a question of further development of such accelerated tests.

The additional investigation of the copper-containing aluminium-magnesium alloys in the absence of stress was not made, as Dr. Champion assumed, to investigate the influence of the stress, but to investigate whether the results obtained by the accelerated stress-corrosion test are confirmed by the standard corrosion test. The influence of stress on the stress-corrosion of an aluminium-magnesium alloy was investigated in a paper by one of the authors.* Generally, stress-corrosion tests were preferred because most of the failures of aluminium-magnesium alloys which occurred in service were caused by stress-corrosion. Dr. Champion recommends stressing the specimens in the stress-corrosion test to a percentage of the initial ultimate strength or proof stress, and in the final series of our investigation, where alloys of different composition and condition were compared by different corrosion-testing methods (Table X), the specimens in the accelerated test (laboratory test 3)

* P. Brenner, *Vorträge der Hauptversammlung 1938 der D.G.M.* (V.d.I.-Verlag).

were stressed to 60% of the 0.2% proof stress. The other stress-corrosion tests were generally made at 10 kg./mm.² for the alloy containing 7% magnesium, and at 15 kg./mm.² for the alloy containing 9% magnesium. We feel that in future investigations it would be much better to determine the whole stress-time curve of each material and not, as is usual, only one point of the curve.

We agree with Mr. Metcalfe and other contributors that accelerated corrosion tests must be applied with great caution. It is emphasized that our experiences are restricted to aluminium-magnesium alloys and cannot be applied to other alloys without further investigation.

The simplicity of set-up and the saving of space brought about by testing a great number of specimens were the main reasons why we stressed the specimens by bending and not by tension. In addition, specimens stressed by bending are expected to fail in a shorter time than those stressed by tension, because the increase in stress caused by crack propagation or general corrosion is much greater in bending than in tension. Theoretically, in the former case the stress increases with the square of the reduction of the thickness of the specimen, and in the latter it increases linearly.

The failure by fracture of a homogenized 7%-magnesium alloy containing copper (Table IV), mentioned by Dr. Quarrell, was caused by a slight tendency to intercrystalline attack in addition to general attack, though the microstructure appeared homogeneous. The duplicate specimen which failed by yielding showed a similar attack and microstructure. This phenomenon was observed in another test with a copper-containing alloy (alloy 5, Table III). One of the un-aged specimens failed by yielding and the other by fracture. It is possible that a limit exists here where very slight differences in the attack (more general or more intercrystalline) lead to yielding or fracture.

Commenting on Table X, Dr. Quarrell said that he is not clear what advantage is gained by using the acid solution. We agree that if the specimens with low corrosion-resistance are compared, the advantage of acceleration is indeed not so great because all these specimens failed in less than 14 days in all four tests. However, the advantage of the acid solution is more clear in the case of specimens with high corrosion-resistance, where, as shown in Table X, a remarkable acceleration is obtained. Specimens with a life of less than 20 days in the acid solution did not fail after 140 days in the other tests.

We agree with Dr. Evans and Mr. Edeleanu on the effect of internal stresses, which depends to a high degree on their direction and distribution, and which may be harmful after ageing, even in the case of compression.

Dealing with the point made by Mr. Murphy and other contributors as to whether there is a correlation between microstructure and stress-corrosion-resistance of aluminium-magnesium alloys, we should like to say that many modifications of the microstructure in the different conditions of heat-treatment and cold deformation exist which could not be described in the paper, and that the micrographs are not always clear enough to show all the details which are visible under the microscope. In Fig. 21 (Plate XX) the precipitate does not form a continuous network, as we understand it, and as it is shown, for example, in Fig. 3 (Plate XVIII), where fine continuous lines are formed at the grain boundaries. The precipitate in Fig. 21 consists of coarse particles which are isolated or form only short isolated lines, so that the corrosion attack finds no continuous path to penetrate along the grain boundaries. In addition, there is a lot of precipitate within the grains which, as we suggest, favours a more homogeneous corrosion and discourages selective attack. This may be the main reason why specimens with a microstructure corresponding to Fig. 21 gave a relatively high stress-corrosion-resistance (33 days in the acid solution).

Dr. Champion has pointed out some discrepancies between the results of micro-examination and the corresponding stress-corrosion-resistance in the acid solution. The results compared are taken from different series which were tested at different times with alloys of different magnesium content (7% and 9%) and different heat-treatment. It may be that the conditions of testing were not quite the same in both series and therefore a direct comparison of the results is not possible. In cases of such contradictions or doubts a final decision, in our opinion, must be made by an additional test under seawater or service conditions.

The results obtained by Mr. Murphy on a 10%-magnesium alloy in the cast state are very interesting. We agree that our results cannot be applied to cast material without further investigation. It is difficult to compare these results because of the different magnesium contents and the different methods of heat-treatment, but if the results of laboratory test 3 with the 9%-magnesium alloy in the un-aged condition (Table X) are considered, the water-quenched specimens have also a higher corrosion-resistance than the furnace-cooled specimens. To explain the contradictory results of the micro-examination, it would be of interest to know whether the slowly cooled cast specimens failed by intercrystalline attack, pitting, or general corrosion.

In reply to the question asked by Mr. Perryman, we suggest that stress might increase the rate of general attack, but our tests give no information on this point because stress-corrosion tests by bending are not very suitable for investigating this effect, which may be investigated more exactly by tension-corrosion tests.

The results reported on p. 165 refer to an aluminium-9% magnesium alloy of high purity, and it seems that there is good agreement with Mr. Perryman's results on the influence of impurities on the microstructure of a 7%- and a 10%-magnesium alloy. The same seems to be true for his investigations of the influence of small additions of zinc, if they are compared with Table VIII. In the quenched condition the alloy containing 0.5% zinc (alloy No. 9) shows a higher stress-corrosion-resistance than the zinc-free alloy (alloy No. 12) up to an ageing treatment of 3 days at 100° C.; at higher ageing temperatures and time the results are reversed. The water-quenched alloy containing 1.1% zinc, on the other hand, has a lower stress-corrosion-resistance, especially in the range of lower ageing temperatures.

The specimens in the partially immersed stress-corrosion test failed mostly at the lower part, at some distance from the fixing point. We suggest that this is caused by a slight increase in the bending stress from the upper to the lower part of the specimens. Failures at the liquid level were relatively few.

Dr. Champion touched on the effect of copper in these alloys. The results with the different additions of copper (0.06% and 0.9%) may be explained by the constitution of the system Al-Cu-Mg. According to the ternary equilibrium diagram at 460° C., given by Little, Hume-Rothery, and Raynor,* an aluminium-7% magnesium alloy containing 0.06% copper is homogeneous, whereas in the same alloy containing 0.9% copper a second phase \bar{S} (corresponding to Al_2CuMg) occurs. If Figs. 9 (a) and (b) on p. 171 of the present paper are compared, it is obvious that in the room-temperature-aged condition (a), the one-phase copper-free alloy has a much better corrosion-resistance than the two-phase copper-bearing alloy. In the artificially aged condition (b), the corrosion-resistance of the copper-bearing alloy is almost unchanged, because no phase change has taken place, but the corrosion of the copper-free alloy is much increased as a consequence of the decomposition of the solid solution. Since the alloy with the lower copper content (0.06%) is homo-

* A. T. Little, W. Hume-Rothery, and G. V. Raynor, *J. Inst. Metals*, 1944, 70, 491.

geneous, its corrosion-resistance is not much different from that of the copper-free alloy (alloys 4 and 5, Table III). The other alloys were copper-free: this means that no copper could be determined by chemical analysis.

To the contribution by Dr. Siebel and Dr. Vosskühler we may reply as follows:

We also observed a slight decrease in the mechanical properties after furnace cooling (Table VIII). Since, however, the figures were still high, we did not attach any significance to this feature.

Regarding the correlation between microstructure and corrosion, contrary to Dr. Siebel and Dr. Vosskühler we have observed the following:

(1) There was always a distinct correlation between microstructure and intercrystalline corrosion.

(2) A microstructure which was not completely stabilized by heterogenization was always susceptible to stress-corrosion after ageing.

(3) Material with isolated particles precipitated at the grain boundaries always had good stress-corrosion-resistance, especially if additional precipitation occurred within the grains.

(4) A fine continuous film of precipitate at the grain boundaries always led to intercrystalline corrosion.

It is true that the constituent Al_3Mg_2 is attacked by hydrochloric acid, but not by alkalis. In spite of this fact, the type of attack was very similar in both the acid solution and the sea water. The reason for this phenomenon is perhaps that the corrosion of aluminium-magnesium alloys is mainly an electrochemical process, in which the electrical conductivity, which is increased by the addition of acid, is an important factor. It would be worth-while making further investigations in this field.

We agree with Dr. Sutton that the precipitation of small amounts of β or β' from solid solution exceeding the equilibrium is facilitated by strain and may frequently be the cause of intercrystalline corrosion in service. If the material is used in a condition in which all the magnesium in excess of that in solid solution at room temperature is precipitated from the crystals, as produced by furnace cooling, this danger is much reduced, because no additional precipitation can later take place under service conditions. As shown in Tables IX and X, even cold reductions of 15–20%, followed by 3 days' ageing at 100° C., do not reduce the stress-corrosion-resistance seriously in the case of furnace cooling, while in the case of water quenching or air cooling this treatment has a very deleterious effect.

Aluminium-magnesium alloys with higher manganese contents than usual were not investigated because they are very difficult to manufacture, especially alloys containing 7 or 9% magnesium. Manganese tends to form segregations in the form of hard and brittle needles of manganese aluminide (MnAl_6), which impair the mechanical properties.

JOINT DISCUSSION ON PAPER BY DR. P. H. JENNINGS, DR. A. R. E. SINGER, AND MR. W. I. PUMPHREY: "HOT-SHORTNESS OF SOME HIGH-PURITY ALLOYS IN THE SYSTEMS ALUMINIUM-COPPER-SILICON AND ALUMINIUM-MAGNESIUM-SILICON", AND PAPER BY DR. P. H. JENNINGS AND MR. W. I. PUMPHREY: "A CONSIDERATION OF THE CONSTITUTION OF ALUMINIUM-IRON-SILICON ALLOYS AND ITS RELATION TO CRACKING ABOVE THE SOLIDUS." *

(*J. Inst. Metals*, this volume, pp. 227, 249.)

DR. E. G. WEST,† B.Sc. (Member): These papers form part of a long-term programme of fundamental investigations into the welding of aluminium alloys. The researches that are being carried out by the team at Birmingham University have as their general objective the improvement of the welding of existing alloys, with the possibility of developing new alloys of better weldability and, of course, satisfactory other properties. The first factor investigated has been the liability of the alloys to cracking, but other factors governing the weldability of aluminium alloys are also included in the programme.

The present papers mark a further stage in the analytical part of the work, and additional papers will be submitted when what might be called the synthesis stage of the work is ready for publication. Nevertheless, the results to date can give some valuable indications of weldability to the practical welder and engineer. Because I wish to emphasize the practical aspects, I confine my remarks to the first paper, but there are several interesting theoretical questions worthy of full discussion.

It is likely that the results obtained on the aluminium-magnesium-silicon series will be found to have immediate bearing on the welding of these alloys, which are widely used in the composition range up to about 1.5% magnesium and 1.5% silicon. The alloys in commercial use, covered at present by the omnibus specifications BS/STA.7—A.W.9 and A.W.10, vary appreciably in composition, and it may well be that the researches have already progressed sufficiently far for the manufacturers and users of these alloys to choose the composition range which will give greatest freedom from cracking. The effect of other elements, of course, must be taken into account—the impurities such as iron, and additions such as chromium and copper—and the investigators should venture into these quaternary, and higher, fields.

Their results on the aluminium-copper-silicon alloys are particularly interesting, because they largely confirm the work already reported by the

* Discussion at the Annual General Meeting, London, 16 March 1948.

† Technical Director, Aluminium Development Association, London,

British Non-Ferrous Metals Research Association on alloys of the D.T.D. 424 type in both cast and wrought forms.* It was considered that aluminium-copper-silicon alloys might find useful employment for certain applications, but their commercial use in wrought form is not yet accepted. This present work has thrown more light on the fundamental causes of cracking in these alloys and has indicated the useful composition ranges.

In Fig. 1 (p. 234), the ternary "cracking" diagram for the aluminium-copper-silicon alloys, up to about 1% silicon and nearly 4% copper there are two ranges which are marked as showing about 7 in. of cracking in the ring-casting test. The figures in Table I (p. 232), from which these "iso-cracking lines" were plotted, indicate that the 7 in. and the 6 in. areas might very well be made into one, giving a much simpler diagram. Point is given to this by the statement that individual results differed from the mean by $\pm 30\%$ in some cases. Taking this into account a simpler diagram could have been produced. It is also stated that any figures for length of cracking which differed seriously from the mean—any "rogues", perhaps—were disregarded. Could the authors give more information as to their definition of a "rogue" figure?

The authors state that at a certain silicon and copper content there is a change-over from rather coarse columnar crystals to a smaller equi-axed structure in both ring castings and welds. There is no indication of the approximate composition at which this change-over takes place. It would be particularly interesting to know this, as there appears to be some evidence that the finer structure is less liable to cracking than the coarse structure.

MR. R. CHADWICK,† M.A. (Member): Aluminium-copper-silicon and aluminium-magnesium-silicon alloys are, with aluminium-silicon-iron alloys, which the authors have previously dealt with, some of the most important basic systems involved in light-alloy welding, and the data obtained are of great value in explaining welding behaviour.

Furthermore, a knowledge of the hot-short ranges in light alloys is important in almost all branches of metallurgy, and marked hot-shortness, which can be associated generally with a long freezing range, is detrimental not only in casting and in welding, but in hot rolling, forging, and in fact in all kinds of hot manipulation. A sufficient knowledge of the various alloy systems to enable compositions with shortened freezing ranges to be selected is therefore of wider significance than the authors indicate.

I think that it would have increased the value of the paper if the earlier work of Lees on a closely related subject, which the authors mention, had been discussed in more detail. Lees, investigating the behaviour of a wide range of casting alloys while cooling under constraint, showed that a long freezing range, particularly where it was associated with the presence of a small amount of eutectic, resulted in hot tearing, the magnitude of which was correlated with a numerical factor called the "eutectic index". Personally, I prefer the present method of expressing results, rather than the use of such an artificially expressed property, but the conclusions in the two papers are the same. I would, however, like to see the use of the term "hot tearing" continued in its application to castings. This terminology is more fully descriptive than the present one of "hot-shortness", and its use would have preserved uniformity and continuity in this subject.

* J. Pendleton and E. A. G. Liddiard, Paper presented to the British Welding Research Association Symposium on the Welding of Light Alloys, October 1946, and published in *Sheet Metal Ind.*, 1947, **24**, (246), 2062; (247), 2273.

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I agree with Dr. West about Fig. 1. With the method of investigation used there is not much difference between a 6 in. and a 7 in. crack, and, in the absence of other evidence for the two separate hot-short areas, it would have been better not to indicate the presence of more than one.

I notice that the authors refer to some work by Tichy in America on the recovery of Duralumin from aircraft scrap. Similar work was carried out by the B.N.F.M.R.A. many years earlier and perhaps this could have been referred to. The B.N.F.M.R.A. also carried out work on the welding of aluminium-copper-silicon alloys, and it is interesting to observe that, starting from a different point of view, they arrived at a similar conclusion with regard to the most suitable alloy for welding—that is one containing copper and silicon, in which the silicon content is roughly twice the copper content.

The second paper, while showing in some degree the relationship of earlier results to the known equilibrium, does not really establish the fundamental relationships. For example, there is no apparent reason given for the absence of a true solidus with a preponderance of iron, though behaviour in the welding experiments confirms the observed freezing curves. Nor is there any real explanation of the isolated land-locked pools of eutectic which, instead of being present, as one would expect, between the primaries, are actually inside the primary grains.

It is interesting to go a little further in comparing the results of the work on aluminium-silicon-iron alloys with actual welding experience, and especially the effect of added manganese. It is, for example, well known that when using unalloyed aluminium filler rod the iron should be present in greater proportion than the silicon. When manganese is present in the filler rod it combines to form a stable compound with the iron, and decreases the ratio of free iron to silicon, causing an increase in the hot-short range; the manganese-containing alloy is therefore unsuitable as a filler rod. Manganese is, however often present in the basis metal, where its grain-refining action counteracts the effect of increased freezing range. I should like to warn the authors, therefore, that when they come to deal with more-complex commercial alloys, which often contain manganese, equilibrium is likely to be considerably changed, while in addition other considerations may be significant besides the freezing range.

Finally, I would like to raise an important point in regard to the method of presentation adopted in reviewing earlier work, and this concerns the diagrams reproduced in the second paper. It is most undesirable to continue the use of the original symbols adopted by authors over the last 20-30 years for the various phases, when there is now a definitely established and recognized system. The paper would have been much easier and clearer to read if, having stated, in Table I for example, the symbols used by the earlier authors, the present authors had, in their subsequent Figures, used a standard terminology. As it stands, it is most difficult to compare these Figures, for particular phases have different symbols in each diagram, and single symbols are used in different diagrams to represent entirely different phases. Most recent publications have employed the terminology used in a series of papers to the Institute of Metals by H. W. L. Phillips, and the present authors would be well advised to follow this example.

MAJOR P. C. VARLEY,* M.B.E., M.A. (Member): I have read these papers with great interest, and must congratulate the authors not only on the manner in which they have carried out their experimental work and presented their results but also on the very considerable extent to which they have been able to account for their observations on theoretical grounds by reference to

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published constitutional investigations on the various alloy systems which they have studied.

Since in so many of the systems examined the ring-cracking and welding tests agree so well with the published diagrams, it is puzzling to find that there are marked exceptions to this rule, and that even in the comparatively simple case of the aluminium-copper binary system there is a marked anomaly, namely the second maximum in the hot-shortness curve, to which the two previous speakers have referred. In this case the constitutional diagram is well established, and it does not seem possible to explain the phenomenon on straightforward constitutional lines, since the diagram is such a simple one in this region. I am therefore forced to the conclusion that some other factor or factors must be operating.

There seem to be two possibilities, one of these being the presence of variable amounts of gas dissolved in the metal, either as the result of unintentional variations in the preparation of the alloys or, more probably, due to changes in the solubility relations as a result of variations in the copper content. Some work by Lees * showed that gas content could have an effect on hot-shortness, though the results obtained by Lees were rather variable from one system to another. My own experience in this direction has been confined to the behaviour of the metal during working at temperatures just below the solidus, where it is found that gas porosity may have a disastrous effect on ductility.

The other possibility is that the inevitable introduction of a small quantity of sodium from the flux used in the melting operation may be responsible for some of the discrepancy. Sodium has long been held to be responsible for poor ductility at high temperatures of alloys of the Duralumin and M.G.7 types, though I feel that in many instances it is a case of giving a dog a bad name.

With regard to the two ternary systems, the aluminium-copper-silicon system is fairly straightforward, since the constituents present are merely those of the two component binary systems. Apart, therefore, from the difficulty with the straight copper alloys already mentioned, there are no complications. The aluminium-magnesium-silicon system, on the other hand, is not so simple, owing to the presence of the compound Mg_2Si . The problem has been discussed at some length by the authors, and they have been able to account for their observations in a general sort of way, but the agreement is not exact. For example, the ridge in the cracking diagram does not correspond exactly with the quasi-binary aluminium-magnesium silicide line, though it is fairly close to it up to about 3% magnesium. If the authors' explanation is correct, one would expect the points of maximum temperature of the secondary separation of Mg_2Si to have more significance with regard to hot cracking than the quasi-binary line, but the ridge in the cracking diagram corresponds with this line only at the point where the two cross.

Again I am forced to the conclusion that other factors are operating, and that the simple concepts of freezing range and proportion of eutectic liquid available for healing are insufficient to explain all the observations when more complex systems are under consideration. It may be necessary to allow not only for the varying proportion of liquid present at different temperatures, as is suggested by the authors, but also for variations in the coherence of the alloys above the solidus due to varying constitution at different temperatures.

As an extreme example of this, I would mention an observation made by Dix, Fink, and Willey † in 1933, and discussed by Phillips.‡ If an alloy with

* *J. Inst. Metals*, 1947, **73**, 537.

† *Trans. Amer. Inst. Min. Met. Eng.*, 1933, **104**, 335.

‡ *J. Inst. Metals*, 1943, **69**, 275.

8% manganese is cooled it begins to solidify at about 760° C., and becomes more and more pasty as the temperature falls, until at 700° C. it is almost solid. Just below 700° C. a peritectic reaction takes place and the alloy becomes almost liquid again. Such an alloy might well crack badly at or above 700° C., and yet the cracks would heal completely at lower temperatures.

With regard to the second paper, on the aluminium-iron-silicon alloys, the authors have already quoted my views in several places, and I have nothing further to add which might help our understanding of their observations.

Thus, while these papers represent a very considerable advance, and provide a firm foundation for the prediction of hot-shortness behaviour in simple alloy systems from constitutional data, there still appear to be some gaps in our knowledge, particularly when the more complex systems are under consideration.

MR. R. W. RUDDLE,* M.A. (Member): During the discussion on the previous papers in this series on the hot-shortness properties of aluminium alloys, I, in company with several other speakers, voiced some doubt as to the satisfactoriness of the tests employed; in particular, I was not at all happy about the method of assessing the susceptibility of the different alloys by means of the length of cracking observed in the ring castings and welds. I feel, therefore, rather disappointed that the authors have not considered this matter further in the present papers, and have not done anything to make quite sure that they are not being misled by the indications of the tests and their method of assessing the test results. Possibly they have carried out some checks of this nature since the present papers were submitted for publication. If this is the case, I hope they will say so in their reply to the discussion.

My doubts of the ring-casting test have, in fact, been somewhat increased by what I have read in the paper by Jennings, Singer, and Pumphrey. It seems to me that a test which, as is stated, it is necessary to reproduce 16 times on occasion, and in which variations in the length of cracking of up to 30% occur, can scarcely be regarded as satisfactory. Severity of cracking is something which is extremely difficult to judge; furthermore, it may not be of great importance in practice. From the practical point of view, what is important is whether the casting or weld cracks at all, and I should like to re-affirm my conviction that any test of hot-shortness should be of the "crack or no-crack" variety.

It is important that a test of hot-shortness should give reasonable agreement with practical experience in industry. I do not think that this is true of the ring-casting test. For example, the authors' results indicate that an alloy containing 10% copper is virtually free from hot-shortness, but I do not think that this is borne out by the behaviour of these alloys in the foundry. The Light Metal Founders' Association † grade 3L8 alloy, which contains 13% copper, as "B", "A" being the highest possible rating and "C" the lowest. The authors' result is, moreover, not in agreement with the work of Lees,‡ who found that an alloy containing 17.1% copper possesses some tendency towards cracking.

Again, the present authors state that, in the aluminium-magnesium series, the danger of cracking is negligible at magnesium contents of over 8%, but, according to Lees's results,‡ an alloy containing 10.9% magnesium is not entirely free from hot-shortness, while the Light Metal Founders' Association

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† Light Metal Founders' Association, "Light-Alloy Castings—Some Notes on the Relationship of Material and Design". Booklet: 1944.

‡ *J. Inst. Metals*, 1946, 72, 343.

booklet places D.T.D. 300 alloy in "B" class. Like the authors, I find it rather strange that the aluminium-magnesium alloys exhibit only a small tendency towards hot-shortness, and I wonder whether in this instance the ring-casting test may not be misleading. It may be noted that Lees's results indicate that an alloy containing 5.6% magnesium had relatively poor hot-shortness properties.

I think that the general picture afforded by the authors' tests is qualitatively correct, but I feel that some caution should be used in applying the results of these tests in industrial practice. In particular, some alloys which, according to the test results, are not hot-short may in fact have some tendency in this direction.

The authors seem quite adequately to have explained their results on theoretical grounds, and they should be congratulated upon this. One point which, however, seems a little obscure is the reason why aluminium-copper alloys containing about 2% copper show less hot-shortness than alloys containing either 3% or 1%. A possible explanation which occurs to me is that the second peak in the cracking-composition curve is caused by a change in the mode of solidification from columnar to equi-axial crystallization. It is to be expected that in general an alloy solidifying to form equi-axial crystals will show more hot-shortness than one forming columnar crystals, the reason being that in the latter case a tendency towards the formation of a completely solid outer shell exists. This outer shell should possess considerable ductility, and should be able to accommodate the imposed constraints without cracking.

In the first paper, the authors give some figures for the so-called "effective solid-solubility limit" and state that these limits have been assumed to coincide with the composition of maximum hot-shortness. This assumption is, I feel, unwarranted and is, in a sense, putting the cart before the horse. Surely, the only correct way of determining the composition at which the intermediate constituent first makes its appearance under the casting conditions used is by micro-examination? It seems to me that the authors' case would have been greatly strengthened if they had done this.

I have read the second paper with much interest, and I am very glad to see that the authors have been able to find an explanation for the effect of iron in inhibiting tearing in aluminium-silicon alloys. The explanation offered, namely that the "effective solidus" temperature falls from 615° to 577° C. at an iron : silicon ratio of 1 : 1, is probably correct. I use the term "effective solidus" because it is clear that the actual solidus occurs at 577° C. in all cases, since some free silicon is always present. The validity of the explanation, therefore, clearly depends on the disposition of the residual liquid. The authors state that Major Varley has observed that, in alloys in which FeAl_3 is present, this residual liquid is in the form of pools within the crystals, but they produce no evidence to show that this holds true in their ring castings. It is, I feel, a pity that the authors have made no attempt to prove their point by microscopical examination, which they could easily have done, for, lacking such proof, the validity of the explanation must remain in doubt.

MR. E. A. G. LIDDIARD,* M.A. (Member): Others have already pointed out that the idea that there was a correlation between hot tearing in castings and cracking in welds has been the guiding principle of earlier investigations by the British Non-Ferrous Metals Research Association. The difficulty there lay in devising tests which would indicate such correlation if it existed. The work that the present authors have done is of very considerable interest

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to all those who have worked on the problem, and their surprisingly close correlation between casting hot-tearing and restrained-weld-cracking tests is a matter for congratulation. At the same time I cannot help feeling that the deviation which they get in the ring tests does not allow them to draw the diagrams which they give in the paper with any degree of confidence.

The ring test, however, is extremely interesting; and, since imitation is the sincerest form of flattery, we have done a certain amount of work using this ring test, together with other tests, to assess casting characteristics, notably those used by Lees. I am afraid that the reproducibility of our results on the ring test was not even as good as that claimed by the authors. I hope that the authors will be prepared to hand on to us, by personal instruction, the means of getting the necessary reproducibility. That is a very genuine attempt to get an invitation out of them!

In the restrained-weld test, I should be interested to know whether the authors ever get cracking along the side of the weld rather than along the centre. In one of their earlier papers they showed a photograph with the crack going right down the centre of the weld, but in the work which was done at the British Non-Ferrous Metals Research Association we often obtained cracks not down the centre of the weld but at the weld junction. A crack down the centre of the weld could, of course, be explained by the hot-tearing mechanism, but the weld-junction crack is rather more difficult to explain in that way. One can regard a weld as a small open-top chill casting where solidification takes place from the walls, which are the edges of the original basis metal. The columnar crystals will probably grow towards the centre, and the tendency, if there is a restraint during solidification of the weld, will be for cracks to occur down the centre. At the side of the weld, however, one would expect the material of the weld junction to be solid before the metal in the weld was coherent, so that it is very difficult to see how weld-junction cracking, which is very common in practice, can arise from hot tearing.

Of course, weld-junction cracking may occur at well below the solidus temperature, and might be caused by oxide films at the junction, or lack of welding, or it could be due to some general weakening of the basis metal as a result of incipient fusion and consequent gas evolution. The latter is particularly likely to occur in the aluminium-magnesium alloys. Such weakened areas may tend to crack on solid contraction, but one would not expect any correlation between that type of cracking and a casting hot-tearing.

Weld-junction cracking might be brought about by a similar mechanism to hot tearing, either because there is inverse segregation of a low-melting-point constituent to the junction, i.e. the original walls of the chill, or, possibly, when the solidus temperature of the basis metal is appreciably below that of the filler metal which is in the pool. In both cases, however, one has to postulate a very flat temperature gradient, since weld-junction cracking will not occur by hot tearing in the presence of a steep temperature gradient from the original basis metal to the centre of the weld pool.

It is very important, therefore, that in any work which is done on the correlation between welding characteristics and casting characteristics the location of the crack in the restrained-weld test should be noted.

I should like to say a word on the question of the freezing range and the distribution of the last-melting-point constituent. Mr. Chadwick said that the freezing range was itself extremely important. I agree with this and so, of course, do the authors; but the distribution of the lowest-melting-point constituent is also important, as the authors suggest in their second paper. In some work that Lees did in explaining the comparatively high resistance to hot tearing of D.T.D. 424, he showed that there was a constituent which remained molten at quite a low temperature, but the constituent was found by

microscopic examination to be in globular form and not in a form which would adversely affect the hot-tearing characteristics.

MR. W. A. BAKER,* B.Sc. (Member): Several of the earlier speakers have touched on points which I should have liked to raise. I think that the investigators are to be congratulated on these two papers, because in my opinion there can be no doubt that they extend our understanding of this phenomenon considerably, and they must be a great help to those whose desire it is to develop alloys which are free from this troublesome hot-shortness or hot tearing, whichever expression one chooses to use.

I do not congratulate them unreservedly when I say that, however, because, like an earlier speaker, I have some misgivings about this ring-casting test. I can see that it has obvious advantages; one has merely to make a series of castings in a range of alloys and then measure the length of crack. I seriously suggest, however, that the better way to assess this casting characteristic is to vary the degree of restraint on the material while it is solidifying, and then see whether or not it cracks. I agree with an earlier speaker that the real criterion is "crack or no crack".

In Figs. 1 and 2 of the first paper, the authors show their results on the hot-tearing tendencies of aluminium-copper-silicon alloys. I was particularly interested to see these results, because these are high-purity alloys, and, as Mr. Liddiard has indicated, the British Non-Ferrous Metals Research Association has done a good deal of work on this type of alloy, both in respect of its casting behaviour and in respect of its welding behaviour; but the alloys which we examined were of commercial purity, containing appreciable amounts of iron and manganese and so on. Nevertheless, the results which we obtained—those, for example, published by Lees,† and those by Pendleton and Liddiard for wrought material ‡—check very well indeed with the results in this paper. Incidentally, the welding behaviour of these aluminium-silicon-copper alloys was thought to be so favourable that the Association patented them (B.P. 575,589) for that application.

Turning to the interpretation of this behaviour of the aluminium-silicon-copper alloys, on p. 243 the authors suggest that the favourable effect of substantial additions of silicon to the copper alloy is due simply to an increase in the proportion of the ternary eutectic phase; at least, that is the way in which I interpret what they say. I am not satisfied that this is the whole story. If you take a cooling curve from this particular alloy, you find, as one of my colleagues has done, that the aluminium-rich primary solid solution begins to separate at 618° C., and then at 565° C. or thereabouts a very substantial proportion of silicon complex separates at an approximately constant temperature. Mr. Liddiard referred to this earlier, and I am merely enlarging on the point. Finally, the alloy solidifies at about 512° C. If you quench samples at intervals between the second and final arrest, you find that the silicon complex has separated in such a way that it forms a continuous solid skeleton, leaving the residual liquid trapped in small pools. It follows, therefore, that this small amount of residual liquid, from which the ternary eutectic is formed, does not control the tendency of the alloy to tear.

That brings me to one more point. On p. 242 the authors have estimated the volume of eutectic in those alloys which are virtually immune from tearing, that is to say, the minimum amount of eutectic required to obtain that property. They give figures for binary alloys of the order of 23% by

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† *J. Inst. Metals*, 1946, 72, 343.

‡ *Sheet Metal Ind.*, 1947, 24, (246), 2062; (247), 2273.

volume. Two earlier papers by Singer and Cottrell gave figures of the same order. Lees's tests gave figures of the order of 40% by volume. The interesting point is that if one considers a 3%-copper, 5%-silicon alloy the proportion of ternary eutectic, according to Lees, is only about 10% by volume, and so far as I can see the alloys should tear from that consideration alone. I think, therefore, that it is the separation of the larger proportion of binary silicon complex (20-30% by volume) which is the more important factor. Incidentally, it is worth adding that if one takes cooling curves from a variety of these complex industrial casting alloys, one commonly finds secondary arrests of one sort or another. I suggest that they are very important and should be taken into consideration in any study of casting or welding characteristics.

DR. E. SCHEUER * (Member): The work of the authors seems to me to be very important and interesting. Their method, however crude and disputed, has the advantage of giving for the first time a figure indicating the tendency of an alloy to hot tearing. This figure can be plotted against composition and the results are in agreement with the theoretical reasoning to such an extent that they probably have a considerable amount of physical significance. There is certainly a lot of work to be done before the reproducibility and reliability of the test is developed to a degree that makes it possible to use this test as a standard test in foundry laboratories, but I hope this will be achieved in due course.

The theoretical explanation which the authors give has one advantage over the theory of the eutectic index as used by Lees, because it brings more into the foreground the length of the freezing range, which is obviously very important. It also eliminates the formal difficulty in the theory of the eutectic index, that the eutectic index does not make any allowance for the freezing of alloys with high solid solubility and no eutectic.

There is one point where I would defend Mr. Lees. He has mentioned the fact that very pure metals do not show hot-shortness.† A picture which I published in 1935‡ and which was published in the form of a diagram by Sachs and myself in 1933§ showed cracks developing in a series of aluminium alloys containing between 0 and 80% zinc and between 0 and 15% copper. The pure aluminium did not show any hot-shortness, whereas there was hot-shortness in the alloys. The percentage volume of eutectic has been measured microscopically, and the maximum hot-shortness seems to occur in alloys containing about 1% by volume of eutectic, which is very different from the approximate 20% found by the present authors in their experiments.

Secondly, I want to direct attention to one feature of the process of solidification which is not generally appreciated. Calculation of the change in the relative proportions of solid and liquid metal with temperature during the solidification of alloys gives the results shown in Fig. A. It is possible to calculate the amount of liquid present at the various temperatures during the freezing interval, and this has been done in the upper row of diagrams for various compositions in a simple eutectic alloy system. The black part is the liquid and the white part the solid. The first diagram shows the amount of liquid at various temperatures in the freezing range in an alloy containing 1% eutectic, the second in one containing 10%, and the third in one containing 50%. It will be seen that the bulk of the primary solidification occurs in a very narrow temperature range near the top. From there on the amount of

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† D. C. G. Lees, *J. Inst. Metals*, 1946, **72**, 343.

‡ *Metallwirtschaft*, 1935, **14**, 343.

§ G. Sachs, "Praktische Metallkunde". Berlin: 1933.

liquid is small, down to the eutectic point. As the percentage volume of the eutectic increases, this channel (as I will call it) not only becomes wider but also opens up much more quickly.

It is obvious that the length of the temperature interval over which a small amount of liquid is co-existent with a large amount of solid, is significant for the amount of hot cracking, because any stress set up can rupture the metal during this whole temperature interval. It is not really necessary for the quantity of eutectic to be sufficient to form continuous films round the primary crystals. At temperatures somewhat above the eutectic point, the amount of liquid will then be high enough to produce this effect. For instance, the points marked X and Y in Fig. A show the temperatures between which the

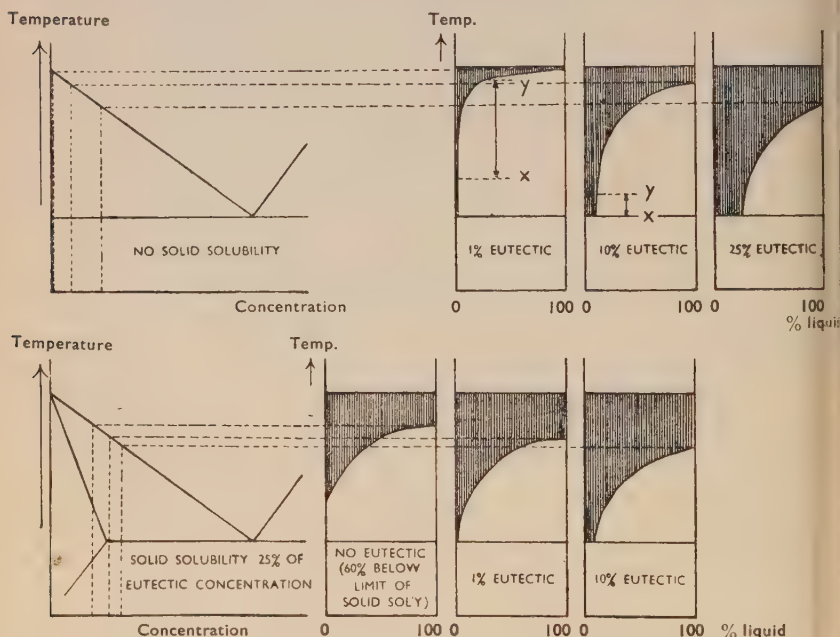


FIG. A.—Change with Temperature of Ratio Liquid : Solid in Freezing Interval.

amount of liquid in this alloy changes from 15 to 1.5%. This explains why even very small amounts of eutectic can produce pronounced hot-shortness, as observed by Lees and Sachs and myself.

In the second row, the same calculations have been made for a system with a limited solid solubility. In this case, wherever one takes the composition, there is never such a narrow channel. The amount of liquid increases very much faster with increasing temperature, and even where there is no eutectic the conditions are never produced which I consider to be most important from the point of view of hot-shortness. We may have here an explanation of the fact that the binary aluminium-magnesium alloys do not show the amount of hot-shortness which they should show according to the freezing interval.

Coming to my third point, in Figs. B, C, and D the general conclusions of

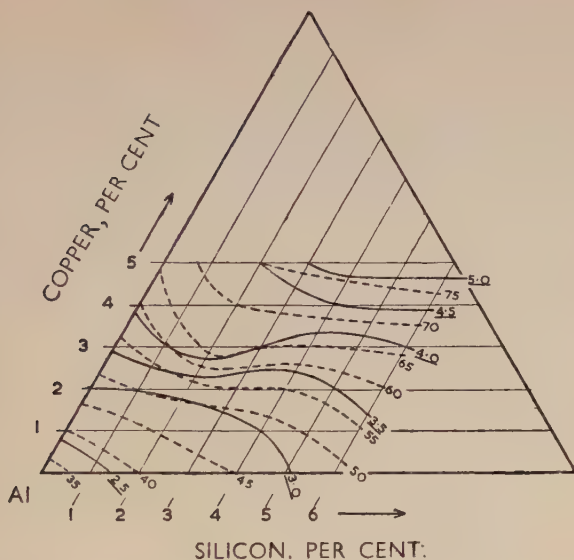


FIG. B.—Change of Mechanical Properties with Composition in Cast Aluminium-Copper-Silicon Alloys.

— 0.1% Proof Stress, tons/in.²
 - - - Brinell Hardness.

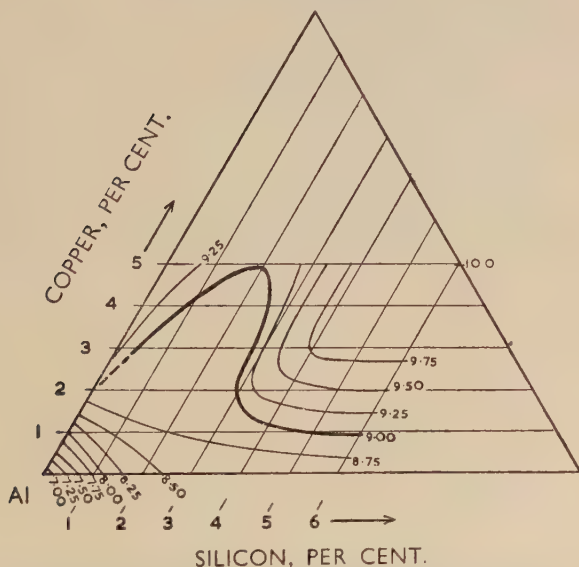


FIG. C.—Change of Mechanical Properties with Composition in Cast Aluminium-Copper-Silicon Alloys. Ultimate Tensile Strength, tons/in.²

the authors regarding the castability of the alloys are borne out to a certain extent. An investigation was carried out in the laboratories of International Alloys, Ltd., dealing with tensile strength, proof stress, and hardness in alloys of the copper-silicon-aluminium system containing some impurities which were kept strictly constant during the experiments. The point of interest here, so far as concerns the present discussion, is the comparison between the diagrams for tensile strength on the one hand and those for proof stress and for hardness on the other. The hardness and proof-stress figures given here show

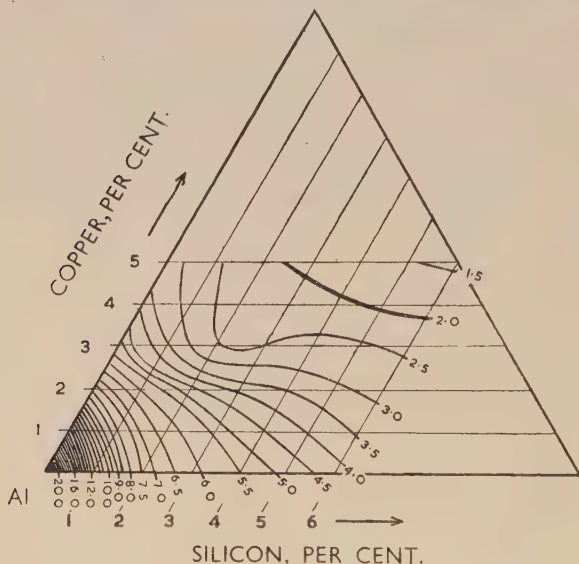


FIG. D.—Change of Mechanical Properties with Composition in Cast Aluminium-Copper-Silicon Alloys. Elongation % on 2 in.

that the increase in copper and silicon content produces a steady hardening effect in the range investigated, but the tensile strength rises in two separate steps; there is first a fairly steep rise, then a sort of plateau where the tensile strength remains fairly constant, and then it rises again. The zone between the two steps we should be inclined to consider as one where the castability of the alloy is comparatively bad. It may not be hot-shortness; it may be intercrystalline shrinkage; but it is something which is connected with the co-existence of a small amount of liquid and a large amount of solid over a considerable temperature range.

DR. P. H. JENNINGS (*in reply*): I have been very interested in much that has been said in this discussion. I was pleased to hear from Dr. West that the work which we have done agrees with the work done by the B.N.F.M.R.A. We have, of course, been in contact with the work of that Association, but we were not able to refer to it in the paper as it has not been made generally available. The chief point made by Dr. West, and referred to by several speakers, concerned the ring-castings diagram of the aluminium-copper-silicon alloys. Dr. West suggested that the two peaks in the diagram should be joined up, and pointed out that the difference between the peak and the trough was considerably less than 30%—the figure which we mention as the

limit of accuracy of the test. That figure relates, however, to one single test, and when many tests are done the average obtained is reproducible within much narrower limits.

I should also point out that the two peaks occur on the welding diagram as well as the casting diagram, though admittedly in slightly different places. We originally chose a much more limited range of alloys than that which was eventually covered. It was for the purpose of finding out whether the two peaks really did exist that we added a number of intermediate alloys, and they did confirm the original findings.

Dr. West also enquired about the position of the change from a columnar to an equi-axed structure. That change took place in the aluminium-copper alloys at 5%, in the aluminium-silicon alloys at 2%, and in the aluminium-copper-silicon alloys at a composition indicated by drawing a straight line across the ternary diagram from 5% copper to 2% silicon.

Coming to the remarks of Mr. Chadwick, I should emphasize that the freezing range is by no means the same as the hot-short temperature range, and the proportion of eutectic is a very important factor in the relation between those two functions. I am grateful to Mr. Chadwick for his amplification of many of our remarks. I agree that there is a considerable amount of confusion and lack of precision in the nomenclature, and in future papers we shall try to clarify the matter and omit the terms "hot-shortness", "hot tearing", and "hot-short temperature range".

In referring to the aluminium-iron-silicon alloys, Mr. Chadwick mentioned the grain-refining effect of manganese in the filler rod. We have been doing some experiments by adding titanium to the filler rod and observing whether that has any effect in decreasing the amount of cracking. The results of that work will be published in due course.

Major Varley referred to the hump in the aluminium-copper binary alloys, and suggested that this might be due to the presence of gas or to the introduction of sodium. We were careful to standardize the experimental conditions, and in the absence of any evidence of a change in the solubility of gas in the binary alloys I cannot imagine that the introduction of gas could have played any part in causing the results which we obtained. We have not made any detailed analyses of sodium content in this particular work, but analyses were carried out on the original aluminium-silicon alloys, and the sodium content introduced from the flux was negligible.

Major Varley points out that the ridge in the hot-shortness diagram for aluminium-magnesium-silicon alloys does not coincide entirely with the ridge in the surface of secondary separation. I agree with him that that is so, but we have not any further explanation to offer of that discrepancy at the present time.

Mr. Ruddle questioned the satisfactoriness of the tests, and particularly the ring-casting test. He mentioned that a test of the "crack or no crack" type was more satisfactory. Although the results are, perhaps, easier to interpret, such a test involves a lengthy experimental procedure and in the case of casting tests necessitates the use of several different moulds. The remark of Dr. Scheuer that the ring-casting test permits the tendency to cracking to be expressed as a definite figure, is in our opinion very important.

Mr. Ruddle also mentions that certain alloys, such as a binary alloy containing 10% copper, and also one with 8% magnesium, which we show as having very low hot-shortness, do in practice crack. The relationship between the result of our test and the absolute hot-shortness (if I may use the term) is not necessarily a straightforward one, and that subject will be dealt with in a later paper. It might, however, be mentioned that the ring-casting test was designed to cover an extremely wide range of susceptibility to cracking and at the extreme ends of this range may not be as sensitive to slightly different

susceptibilities to cracking as tests especially designed to discriminate between alloys in these two ranges. The ring-casting test is, however, very suitable for distinguishing between the susceptibilities to cracking of the great majority of aluminium alloys during casting and welding.

Mr. Ruddle said that no proof had been given for the distribution of silicon-rich liquid in globules in the aluminium-iron-silicon alloys. We have relied on the work of Major Varley in putting forward this theory.

I am interested to hear that Mr. Liddiard has carried out tests similar to our ring-casting test, and that he finds that the reproducibility obtained is not satisfactory. We found that it was necessary to standardize testing procedure very carefully in order to obtain reproducible results. That applies particularly to the casting temperature, which we standardized at 100° C., above the liquidus, and we found that a departure of 10° C. or so from this temperature often substantially altered the results obtained.

We did experience cracking at the edge of the weld as well as at the centre, and sometimes both types of cracking occurred in the same weld. I think the cause of the phenomenon is that the thickness of the weld is greater in the centre, and, although the material at this point may be weaker than the material at the boundary of the weld, the load which it can withstand may nevertheless be more, so that cracking takes place at the boundary. There is also the possibility of cold cracking—that is, cracking below the solidus—and a paper is being prepared on the subject of cold cracking which will deal with that point.

Mr. Baker mentioned that an examination of aluminium-copper-silicon alloys, which had been quenched from between 565° and 512° C., revealed that the binary complex possessed a continuous structure. It is quite possible, though we have never found evidence for it, that in certain alloys the separation of the binary complex plays a greater part in determining the hot-shortness properties than that of the eutectic. I should, however, like to know whether Mr. Baker employed a very long soaking time before quenching his specimens, since that might very greatly affect the structure of the liquid portions.

CORRESPONDENCE.

MR. J. W. JONES,* M.Sc. (Member): I am not sure that it is the intention of the authors that the information from this work should be pushed to the extent of suggesting the ring test as an exact control of foundry practice. It is my impression that the work is essentially concerned with the tendency of alloys to crack under conditions of welding. The ring test is a casting under conditions least likely to lead to success, i.e. with the liquid metal in the mould solidifying and shrinking on to an unyielding core. No foundryman would consider making a casting under these conditions. It is the function of a core to collapse under shrinkage stresses, and local cracks tending to appear would be eliminated by feeder heads. The weld is a casting made under these conditions offering minimum opportunities of success, and as a measure of welding properties the authors are to be congratulated on the development of the ring test. The general tendency for the length of crack to be taken as a criterion of the properties to be measured seems to me to be very well established, but more information would be welcome to satisfy such enquiries as:

(1) What is the precision of the method of inspection for the presence of cracks? Is it just visual inspection with the unaided eye?

(2) Have microscopic or crack-detection methods been used to confirm that

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castings judged free from cracks are really free from cracks of microscopic dimensions?

(3) Do cracks sometimes appear at two or more places in a casting, and, if so, how are these results assessed?

The AUTHORS (*in further, written, reply to the discussion and in reply to correspondence*): Mr. Jones raises the question of the applicability of the ring-casting test to actual casting practice, and suggests that it is more appropriate as a test for welding behaviour. Although it is true that the conditions of stress in which a casting solidifies are seldom as severe as those which obtain in the ring test, the same can be said of many tests for other properties. For example, a constructional material which is to be used at a comparatively low stress is tested to the breaking point.

As Mr. Jones points out, welding is a special type of casting, and this is borne out by the close agreement between the results obtained from the ring-casting test and the restrained-weld test. The ring-casting test would seem suitable for predicting the behaviour of an alloy either when cast or when welded. It is possible, however, that the degree of hot-shortness which is tolerable in certain types of casting is high enough to be outside the useful range of the test, and we would not suggest that the test could in every case be used as an exact control for casting practice.

No special methods were used for the detection of cracks, though in cases of doubt a low-power lens was employed. All that is required is a measure of the extent of cracking, and the neglect of any cracks of microscopic size was not found in practice to alter the order of the result. In a test of the "crack or no crack" variety it would, of course, be important not to overlook any cracks at all in borderline cases.

In reply to Mr. Jones's last point, cracking in the ring test frequently occurs in two or more places, as illustrated in an earlier paper.* In such cases, the result is expressed as the sum of the length of all cracks.

Dr. Scheuer raised several interesting points which call for further reply. He has observed that the greatest hot-shortness in the aluminium-zinc and aluminium-copper systems occurs in the alloys containing about 1% eutectic. In the present work it has been observed that the peak in the hot-shortness curves coincides with the first appearance of eutectic in the microstructure, which is also the composition at which the freezing range is longest. Richer alloys are progressively less hot-short, and the amount of cracking in the two tests becomes negligible when the eutectic content reaches a volume of the order of 20%. Dr. Scheuer's maximum at 1% eutectic is therefore in close agreement with the conclusions of the present work.

The diagrams in Fig. A are very instructive, and suggest that the greater the solid solubility the shorter is the temperature range in which hot-short cracks may form. This may, indeed, account in part for the surprisingly low hot-shortness of aluminium-magnesium alloys; and it would be of great interest to carry out high-temperature tensile tests on these alloys on the lines of the work of Singer and Cottrell † on aluminium-silicon alloys, in order to determine the temperature ranges in which hot-short cracking can occur. It is doubtful, however, whether it is possible to relate the hot-shortness of a system of alloys with the tensile strength at room temperature, as Dr. Scheuer suggests for aluminium-copper-silicon alloys. He postulates that the plateau in the tensile-strength diagram is associated with shrinkage porosity, but there seems no reason why a similar plateau should not exist in the diagram of proof stress.

* A. R. E. Singer and P. H. Jennings, *J. Inst. Metals*, 1946, 73, 197 (Plate XIII).

† A. R. E. Singer and S. A. Cottrell, *J. Inst. Metals*, 1946, 73, 33.

DISCUSSION ON DR. A. LATIN'S PAPER: "PRESSURE AND CREEP TESTS AT CONSTANT HOOP STRESS ON LEAD AND ALLOY 'E' PIPES." *

(*J. Inst. Metals*, this volume, p. 259.)

PROFESSOR E. N. DA C. ANDRADE,† F.R.S. : I am very much honoured by your invitation to open this discussion. The flow of lead was the subject of my first paper, and I have always been very interested in it; but the moment that you start alloying the lead very grave problems arise, of the greatest importance for industry, which I do not think that any of us are really competent to discuss in full; I myself certainly am not. I would remind you of the nature of the formula which is used in this paper by the author :

$$l_t = l_0(1 + \beta t^{1/3})e^{\kappa t}$$

In this formula the constant β is a measure of a transient flow and κ is a measure of a flow linear with the time, or permanent flow, as it is often called. The exponential form of the expression involving κ is a consequence of the fact that the length flowing continually increases : it expresses the relation :

$$\frac{1}{l} \frac{dl}{dt} = \kappa$$

This formula gives a very close fit to experiment over times of flow of some hours, but is probably not exact when very long times are involved, although even here the departures are not large. Mr. A. J. Kennedy is now carrying out some experiments in my laboratory of which the duration is 40 days or more, instead of the hours in my original experiments, and in the later stages of such prolonged flow the transient flow falls somewhat below what is given by the formula, so that it is possible that $t^{1/3}$ should be replaced by some function of the time which, while very close to $t^{1/3}$ up to moderate values of t , drops below $t^{1/3}$ for large values of t , and ultimately becomes constant. However, the formula seems very well adapted for separating the flow into the two components, transient creep and permanent creep, which have a physical significance.

I am not fully aware of the technical application of Dr. Latin's experiments, but I take it that, from the industrial point of view, creep in the lead pipes is not serious as long as it comes to an end, that is, that a pure β creep is not pernicious, but that the κ type of flow is to be avoided, since in the end it is bound to lead to rupture. Perhaps Dr. Latin will tell us what is the ultimate object of his extremely interesting experiments.

With regard to rupture, there is one elementary point that I should like to bring forward for consideration. Whether you get local rupture or not depends on whether, when you begin to get thinning at a particular point, the metal (or other substance which may be in question) hardens or not. You can blow glass because any local thinning is accompanied by cooling and consequent toughening. I think that with lead at, say, 250° C., where there is very small

* Discussion at the Annual General Meeting, London, 18 March 1948.

† University College, London.

work-hardening, internal pressure in a pipe would lead to rapid failure at some point where the walls happen to be initially slightly thinner than the normal. I imagine, therefore, that what is inimical to local rupture is a fairly rapid hardening of the metal, that is, a preponderating β flow. The matter is clearly, in the case of the alloy, a complicated one, partly due to the fact that the concentration of the added metal is probably high in the intercrystalline spaces.

The author considers the fit of my formula to his curves fairly satisfactory, but a very much better fit than that which he shows can be obtained by a careful choice of the constants. I give an example. In Fig. A (Plate XLVIII) the continuous line represents the author's experimental results, as given in his Fig. 9 (p. 274), and the broken line is the curve given by the formula with the values of the constants chosen by him. Mr. A. J. Kennedy has worked out a rapid method of fitting creep curves which will shortly be published, and I asked him to find the constants by this method. He finds $\beta = 6.28 \times 10^{-3} \text{ hr.}^{1/3}$ and $\kappa = 6.7 \times 10^{-4} \text{ hr.}^{-1}$, and points given by the formula with these values of the constants are indicated by the crosses. It will be seen that the fit is then extremely close.

The author speaks of the third stage, that is the stage of accelerated creep, as if it was a real feature of flow under constant stress. This third stage does not, as far as I know, occur when the stress is kept constant unless conditions are such that recrystallization takes place, as it does with certain steels which have been investigated in the neighbourhood of 800° C. We are carrying out some work with lead at temperatures approaching the melting point, and here we shall probably get a final stage of accelerated creep, because at these temperatures there is considerable recrystallization. I should perhaps point out that with a particular lead-tin alloy which I investigated,* there was, at -78° C. , a stage of accelerated flow at certain stresses, but the form of the flow curve was not that usually considered as typical of the "third stage", for the rate ultimately diminished, instead of continually increasing. It is, perhaps, this case that Dr. Latin has in mind when he says that my creep formula is probably not universally applicable. "Universal" is a big word, but, as regards pure metals, close representation of experiment has been verified by me for lead and copper (face-centred cubic), iron (body-centred cubic), cadmium (hexagonal), tin (tetragonal), and mercury (rhombohedral), and by Orowan and Los for aluminium (face-centred cubic). Another point which the author mentions is that when I plotted β against stress I found that it tended to approach a limit, whereas he says that he has never obtained any sign of that limit in his work. However his work lies in quite a different region of stress to that of mine which he quotes: his biggest stresses are just a little less than my smallest ones. The part of my curve of β against stress which lies in the range with which he is concerned is more or less linear, which is what he gets. There is thus no contradiction between our findings.

The solution of many of Dr. Latin's problems is probably to be sought in the relative importance of β and κ flow under the different conditions. Thus in his Fig. 12 (p. 277), comparing pure lead and alloy "E", it will be found that, for the stress which makes $\kappa = 0.0001$ in each case, β is greater for alloy "E" than for pure lead, whereas for the stress that makes $\kappa = 0.0000011$ in each case, β is smaller for alloy "E" than for pure lead; that is, at larger stresses and rates of flow, β flow is more marked, at equal κ , for alloy "E" than for lead, whereas at small stresses and rates, β flow is, at equal κ , more marked for lead than for alloy "E". While this point is not specifically mentioned by Dr. Latin, it seems to support some of his conclusions.

I think that it has been proved that rotation of the axes of the crystallites is associated with the β flow. A point which has not, perhaps, been made

* *Proc. Roy. Soc.*, 1914, [A], 90, 329.

quite clear is that any rotation of crystal planes in the crystallite must be accompanied by slip on those planes, and this slip, as we know, occurs on planes whose separation is very large compared to interatomic distances. This is bound to lead to lack of fit between crystallites at the interface, as illustrated by the model shown in Fig. C (Plate XLIX), where microscope slides are used to simulate glide packets, of varying width. The intercrystalline boundaries thus become full of fragments, whose size is of the same order as the thickness of the glide packets. As Dr. Latin suggests, the κ flow may tend to heal this fragmentation; an ordinary amorphous flow, and interchange of place among atoms as distinct from the slipping on crystal plane which characterizes true crystal flow, will tend to hinder any fracture which is tending to form.

I think that the author's general picture has much to commend it, and I am sure that he and I agree that there is a great deal more to be done before we have a really satisfactory qualitative theory. I am very glad to see him working on the present lines, which I think are bound to lead to most interesting results.

DR. J. McKEOWN * (Member): In most of the previous work which has been carried out at constant stress the stress used was unidirectional, whereas in the work described by the author each element, particularly at the inner surface of the lead pipe, is subjected to a three-dimensional stress system, so that the author was really working under combined stress conditions. I think that it is necessary to recognize this difference. There is a possibility that the presence of the longitudinal stress in particular may influence the values, and also the ratio of the β -flow and k -flow constants. Tests of the type made by the author would be very suitable for an investigation of the effect on the rate of creep and on the extension at fracture of the ratio of wall thickness to grain diameter. In the pure-lead sheaths he tested, this ratio was 6:1, while in the alloy "E" sheaths it was 12:1. The effect of this ratio is likely to be at least as great as the effect of the grain-size itself.

Some work carried out before the war in the laboratories of the British Non-Ferrous Metals Research Association showed that in pure lead the smaller the grain-size the greater the extension at fracture. There are some data on this in a paper of mine.† Thus, in constant-load tests on pure lead having a grain diameter of about 0.9 mm., the elongation at failure remained almost constant with decrease of nominal creep stress, that is, with decrease of rate of creep. In another lead of similar purity, but with a grain diameter of only 0.1 mm., the elongation at failure increased with decrease of nominal creep stress. We are getting some evidence that the same phenomenon may be expected in all lead alloys in tests at room temperature and higher temperatures.

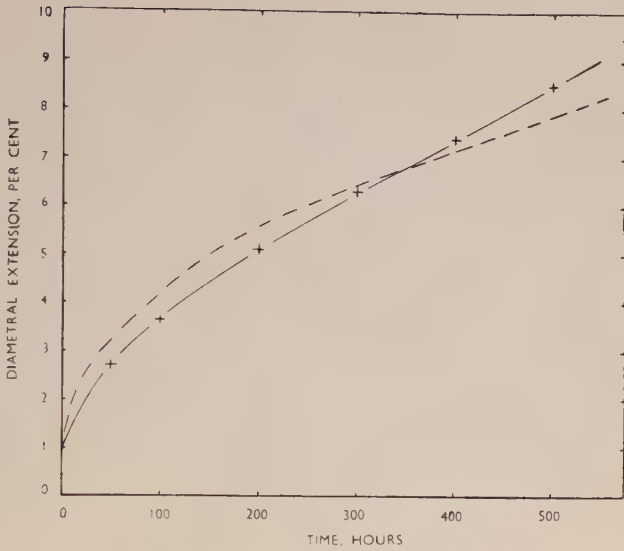
In conjunction with this phenomenon, we have to consider that in any one lead alloy the finer the grain-size the faster the rate of creep in the second stage. We have therefore the two conditions: (1) a fine grain-size giving a rapid rate of creep, coupled with considerable extension at fracture, and (2) a coarse grain-size giving a slow rate of creep, coupled with small extension at fracture. The former condition is suitable for certain applications, and the latter for others.

From this, it appears that in any lead alloy there will be a particular grain-size at which with decreasing rates of creep the extension at fracture will remain constant. With the finer grain-size the extension will increase, and with the larger grain-size it will decrease with decreasing rate of creep.

I should like to ask the author whether any of his constant-stress tests were

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† *J. Inst. Metals*, 1937, 60, 201.


 FIG. A.—Alloy "E", Hoop Stress 1200 lb./in.²

——— Experimental curve.
 - - - - - Calculated curve (Latin).
 + + + Points calculated with
 $\beta = 6.28 \times 10^{-3} \text{ hr.}^{-1/3}$
 $\kappa = 6.7 \times 10^{-4} \text{ hr.}^{-1}$

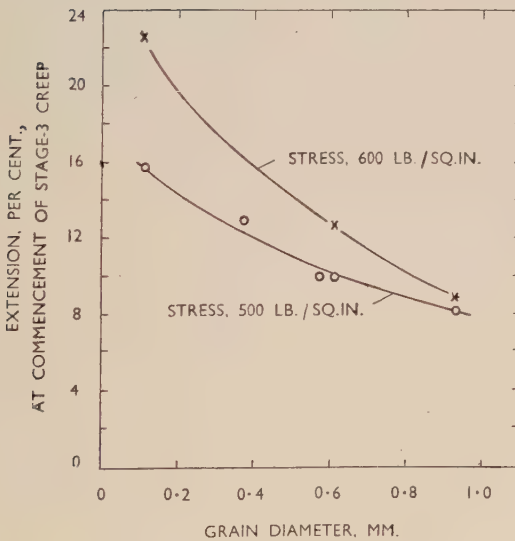


FIG. B.—Effect of Grain-Size of Pure-Lead Specimens on Extension at Commencement of Third Stage of Creep.

[To face p. 670.



FIG. C.—Tension in horizontal direction.



FIG. D. $\times 2.5$

carried to fracture in the case of alloy "E" and, if so, whether the extension at fracture decreased with decrease in stress, as occurred in the constant-load tests.

On p. 284, talking of alloy "E", the author makes the statement that "The greater extension of the alloy, as compared with pure lead, would then appear to be logically accounted for by the retardation, in the harder material, of the start of the final stage-3 local extension, which precedes failure". Has he considered the effect of grain-size on the initiation of the third stage? My own observations, in the case of pure lead, are that the finer the grain-size the greater the extension at the beginning of the third stage of creep. This is shown in Fig. B (Plate XLVIII), where the extension at the beginning of the third stage is plotted against the grain diameter, and it will be seen that the extension at the beginning of the third stage decreases with increase in grain-size.

DR. J. C. CHASTON,* B.Sc., A.R.S.M., A.Inst.P. (Member): Although this paper is greatly to be welcomed as a serious attempt to throw light on the mechanism of creep, I feel that too little care and attention have been given to the experimental approach to the problem. As I understand it, the point which the author seeks to explain is that reported in the third paragraph on p. 263 in the following words: "Briefly, the most important result obtained from these tests is that, whereas the extension obtained with alloy 'E' is considerably greater at high rates of extension than with pure lead, a marked decrease occurs with alloy 'E' when the rate of extension is less than 0.1%/hr." The evidence for this is given in Fig. 3 (p. 266). This Figure gives the test results of some scores of tests on lead, but it is noteworthy that the author does not plot a single test result for alloy "E", but shows simply a dotted line which he asks us to accept. In view of the fundamental importance of that dotted line to the whole of the argument, I think that the individual observations on which it is based should have been included. We are entitled also to ask what the amount of scatter was and how many tests were made; and I submit that answers to those questions must be provided before the alleged effect is discussed.

I should like to consider in a little more detail the general question of the value of bursting tests on lead sheath. The author does not show any photographs of the bursts. In a limited experience in doing this class of test (on sheaths made by a ram-type extrusion press and not by a straight-through press) I found † that at room temperature failure always occurred along the seam. Did that happen in the author's test? In our tests, the sheath opened up in a long split, 8 or 9 in. long, which was always located along the seam. These tests were carried on for between 100 and 500 hr. at room temperature. We also did some tests at 110° C. and got a completely different type of failure; instead of opening up as a burst, the sheath just failed at one point. I have always thought that these results indicated that the seam had inherently less resistance to creep, though probably only slightly less resistance, than the bulk of the metal. That is one of the complications in doing tests on sheaths; some of the others have been referred to by the author. There is the lack of uniformity of the sheath walls, because it is impossible to make a really concentric sheath. There is the fact that a sheath must suffer some distortion in handling after extrusion, and is never perfectly circular or perfectly straight or perfectly free from irregularly distributed cold work. Finally, as several speakers have mentioned, it is necessary to take account of the fact that the stress distribution in a lead sheath under internal pressure is complex.

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† *Elect. Commun.*, 1934, 13, 31.

For all these reasons, I think that the real value of sheath-bursting tests is to obtain data on the performance of the sheath in service. They are a check on all the assumptions which must be made in estimating the strength of a fabricated sheath, and as such they are invaluable to cable engineers; but I do not think that without a good deal of further evidence they should be regarded as evidence on which theories of creep failure should be based. In other words, I think that before the conclusions on which this paper is based can be accepted we want a good deal more experimental work, and particularly work on the relationships between the two materials with carefully prepared test-pieces and tests carried out with all the normal precautions which are accepted as necessary in mechanical testing.

On the theoretical side I would like to protest against the use of the term "grain rotation", because I think that it implies something which does not happen. You cannot imagine a multi-sided grain rotating as a whole; it would push all the other grains aside. I do not think that the author implies that this happens, but I should like to see some other term used to mean the apparent rotation which is observed as a result both of slip and of grain-boundary movement.

As a matter of interest, I would point out that it is not correct to say that this grain-boundary movement in failures of this kind has only recently been discussed. It was discussed by Dr. Rosenhain before this Institute in 1926, when he emphasized, to quote his words, "There can be no doubt that it is possible, and actually must happen, that crystals as a whole may have considerable relative movement under plastic deformation".

DR. E. OROWAN,* F.R.S. (Member): As Dr. Latin himself has stated, the most interesting point in his paper is the observed variation of the uniform extension with the rate of creep. If the wall of a tube suffers local thinning along the crack, its failure under internal pressure is essentially the same plastic-instability process as the necking of tensile specimens; adopting the nomenclature used for tensile tests, I shall call "uniform extension" the (circumferential) extension at points far enough from the edges of the crack. According to the author's observations, the uniform extension is very large in alloy "E" at high rates of deformation, but becomes very small at low rates; with lead, there is a fairly irregular scatter, due probably to heterogeneity of the material used.

The author's explanation for the decrease of the uniform extension with the rate of straining, stated in terms that are less dependent on particular hypotheses, is this. He assumes that the rate of atomic re-arrangements at the grain boundaries increases with the creep rate more rapidly than the rate of glide within the grains; the flaws and highly distorted spots at the grain boundaries, arising from the incompatibility of glide in neighbouring grains, are then more rapidly removed by atomic re-arrangements if the creep rate is high. The danger of intercrystalline fracture, therefore, would be less at high creep rates.

I should like to point out that, quite independently from such special circumstances which may have an effect on the uniform extension in creep tests, there is a general and fundamental reason for the increase of the uniform extension with the creep rate in all cases where the deformation can be considered as a viscous flow with a yield point (in the terminology of the rheologists, where the material behaves like a "Bingham solid"). A tensile specimen from a purely plastic material (i.e. a material whose law of deformation is given by a stress-strain curve, with no time effects involved) necks when the maximum load is reached; if one of its cross-sections was initially slightly smaller than

* Cavendish Laboratory, Cambridge.

the rest, it goes over its load maximum before the other sections have reached their maxima, and so further extension is confined to the weakest section. If the true stress-strain curve is given, the maximum-load point on it can be obtained by a well known simple graphical construction; with the stress-strain curves of common metals, the uniform extension (i.e. the extension before necking, which is practically equal to the final extension outside the zone of necking) is usually between 0 and 20%.

What is the corresponding behaviour of a purely viscous material in the tensile test? If the material obeys Newton's law of viscosity, with the coefficient of viscosity η , the extension of a rod of current length x and current cross-sectional area a , under the load L , takes place according to the relationship:

$$\frac{L}{a} = 3\eta \cdot \frac{1}{x} \cdot \frac{dx}{dt} \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$

since the volume $V = x \cdot a$ is constant:

$$dV = x \cdot da + a \cdot dx = 0,$$

and so:
$$\frac{1}{x} \frac{dx}{dt} = - \frac{1}{a} \frac{da}{dt} \quad . \quad . \quad . \quad . \quad . \quad . \quad (2)$$

substitution of (2) in (1) and integration gives the variation of the cross-sectional area in the course of the extension:

$$a = a_0 - \frac{L}{3\eta} t \quad . \quad . \quad . \quad . \quad . \quad . \quad (3)$$

where a_0 is the initial cross-sectional area. Thus, under a given load, the cross-sectional area of a Newtonian viscous tensile specimen diminishes per unit of time by an amount that is independent of the cross-section; if one cross-section in a rod was initially slightly smaller than another, the difference between the two cross-sections remains constant during the test, and there is no tendency to necking at the weaker point until the specimen thins down so that its cross-sectional area becomes of the same order as the greatest initial difference between the cross-sectional areas at various points. In addition to the point mentioned by Professor Andrade (that thinner sections cool faster), this seems to be the main reason why glass can be drawn out or blown, while metals can not.*

The behaviour of metals under creep conditions differs from that of a Newtonian material in that there is a yield point below which practically no deformation takes place; if the yield point is exceeded, the creep rate increases with the stress (in what follows, the creep is assumed to be mainly viscous; transient creep will be regarded as negligible). At very low rates of creep the stress is practically equal to that at the yield point; as far as the instability of the extension is concerned, the specimen behaves then like one of a purely elastic material, and necks after a small uniform extension. On the other hand, if the rate of extension, and thus the stress, is very high, the representative point is so high up on the stress-rate of strain curve that the existence of the yield point can be neglected and the material can be considered as almost purely viscous with, as we have just seen, very little tendency to necking and, therefore, very large uniform extension. Under (viscous) creep conditions, therefore, slow extension should give small, and rapid extension large values of the uniform extension, provided that the material behaves according to the assumptions made. One of these assumptions is that the maximum load is

* Note added in proof.—Since making this statement I have found that the problem of necking under creep conditions has been treated by A. Nádaí and J. J. Manjoine (*J. Appl. Mechanics*, 1941, 8, A-77).—E. O.

determined by necking; if it is due to more or less brittle fracture taking place before necking sets in, the above-mentioned considerations do not apply. In very slow creep tests at relatively high temperatures, the fracture is often due to grain-boundary slide and is not accompanied by substantial plastic deformation within the grains, or by necking; in such cases, slow extension leads to small values of the uniform extension for an entirely different reason.

MR. L. M. T. HOPKIN,* B.Sc., A.R.S.M. (Student Member): In view of the complex nature of the problems of creep, this paper is of considerable interest, as most of it reports the results of creep tests which were performed at constant stress, thereby removing one of the many variables which have added to the complexity of the subject. It is rare that creep testing in industrial laboratories has been performed at constant stress, in spite of its advantages which have been pointed out by Professor Andrade. It is encouraging to know that the industrial producer, as well as the theoretical physicist, desires to have a more fundamental understanding of creep.

It has been observed that with certain continuous extrusion machines the product varies in composition across its cross-section, and the extent of this variation is probably not uniform along the length of an extrusion. This segregation is due to the complex movement of the metal and to temperature fluctuations occurring during solidification in the machine. This fact might account for the observation that creep-rate variation in specimens tested at one stress was greater for alloy "E" than for pure lead. Fig. D (Plate XLIX) shows the etched cross-section of an alloy "E" pipe produced on a continuous extrusion machine. Chemical deposition of black metallic antimony indicates the regions where segregation of this element is most pronounced.

No mention is made in the paper as to whether care was taken to ensure that the materials were not deformed in any way after extrusion. A small amount of cold work, even as small as 1% strain, would affect the resulting creep curve. It is stated that there was no grain zoning in the materials, although this would not be expected unless the pipe had been coiled without quenching after extrusion. Coiling under any conditions would mean that the results of the work were of little value.

In considering the results of the tests at constant pressure, the author states that the so-called third stage of a typical creep curve is due to localized extension of the specimen. Professor Andrade has pointed out that the acceleration of creep rate is merely the consequence of a rise in stress. This increase in stress can be brought about in three ways: by a reduction in the cross-sectional area during the general extension of the specimen; by a reduction in the effective cross-sectional area due to the formation of inter-crystalline cracks; and by localized extension. It might well be that under some conditions, the first two factors give rise to the third stage of creep long before any localized extension has occurred. Examination of the curves for alloy "E" at constant pressure (Fig. 2) suggests this to be true. The curves for stresses of 900 and 1000 lb./in.² each show the presence of a third stage, although the extension at the point of failure has not been plotted.

It may be noted that a constant-stress device can take into account only the tendency for an increase of stress due to the first of these three factors, so that creep curves of all constant-stress tests show a third stage of creep, but of less magnitude than in the case of tests carried out at constant load. In view of the slight evidence of a third stage of creep in the creep curves of specimens tested at constant pressure, it is not surprising that the results of tests at constant stress give much less indication of the third stage, although it must be remembered that many of the latter curves have not been plotted to failure.

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Perhaps failure occurs in the thin wall of the pipe soon after the commencement of either localized extension or intercrystalline cracking. *

The author has observed that the ratio of β/k increases with decreasing stress, and he is probably correct in suggesting that the ratio will be affected by grain-size. It is possible that with a material of small grain-size the ratio will become smaller as the stress diminishes. This is supported by the fact that in the creep curves of fine-grained lead alloys the ratio of the extension occurring during transient creep to the final extension at failure decreases with decreasing stress.

MR. D. McALLISTER *: The author refers to work being carried out in other laboratories of our Company. I have been associated with investigations in one of these laboratories, and would like to support the views expressed in the paper. In particular, we have recently obtained some results on the effect of grain-size which would seem to throw further light on the creep mechanism and afford some confirmation of the hypothesis which is suggested. This work was carried out on alloy "E" strip instead of pipe, using the conventional static-load tensile creep test at a constant load. The strips were extruded in the laboratory at two different temperatures, one being similar to that used for extruding cable sheaths, i.e. 400° F. (204° C.), and the other 200° F. (93° C.). The respective average grain diameters were 0.3 mm. (which is a normal grain-size for cable sheaths) and 0.05 mm. (which is a very small grain-size indeed).

We found three interesting things. The first was that with the large-grain-size strip the extension at fracture decreased with time to fracture, in the same way as with the pipes, or strips cut from cable sheaths, but the extension of the fine-grain strip increased with time to fracture.

Secondly, the creep of the fine-grain material was very much greater at a given stress. Some actual values may be of interest. At a creep rate of 0.5%/hr. both materials failed with a total extension of 40% in a time of the order of 50 hr. Results have so far been obtained down to about 0.01%/hr., the small-grain-size strip then having approximately 70% extension in 2000 hr., and the larger-grain-size strip 20% extension at failure in 1000 hr. The extension on the small-grain-size material has therefore increased from 40 to 70%, and on the material with the more normal grain-size the extension has fallen from 40 to 20% over the same range of creep rate.

In connection with creep rate and stress, it may be mentioned that at 800 lb./in.² the times to failure were 250 hr. for the fine-grain strip and 2000 hr. for the coarse-grain strip.

The third interesting point, which has also been mentioned by the author, is that except for times to fracture of 100 hr. or so, the type of fracture in the coarse-grain strip was intercrystalline, whereas in the fine-grain strip up to about 2500 hr. to failure, the fracture has been knife-edged.

Similar tests have been carried out on pure lead. The difference in grain-size was not so great, and the results on each material were of a similar order. I mention these results because they agree with the author's predictions and perhaps help a little to disentangle the apparently contradictory opinions which have been published from time to time. It seems that insufficient attention has been paid to this question of grain-size in relation to the properties of lead alloys.

MR. E. R. W. JONES,† B.A. (Member): I am particularly interested in one point in this paper which has already caused a good deal of comment, and that

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is the question of the different behaviour of alloy "E" and pure lead in the rate of extension and mean extension. I agree with the author that at a first glance the figures for pure lead plotted in Fig. 3 (p. 266) do not reveal any trend in this direction, but the points are plotted for materials from a number of sources, and it seems to me that the scatter between the materials from different sources has been in point of fact camouflaging the effect. I tested that by the best method which I had (not having the numerical results available) by drawing a horizontal line so that half the points of any one group lay above the line, and a similar line vertically so that half lay to the right and half to the left. If that is done for the five different sources the results are as follows:

4	10	6	5½	1¼	3¼	4	12	3	3
9	4	5½	5	3¼	1¼	12	4	3	3

I suggest that this does indicate a definite trend. There is over all a greater tendency for the points to be in the first and third quadrants rather than uniformly distributed. If one calculates the probability that that will arise if there is no trend, it works out at no more than 5%. That suggests that it is worth looking rather more carefully at the actual numerical values, to see whether the scatter between the different sources has in point of fact masked the same effect as is found in alloy "E".

DR. A. LATIN (*in reply*): It is impossible for me now to reply to all the points raised. One point which has been raised by Mr. Jones, and also by previous speakers, is the question of scatter. The scatter is always large in tests of this type, but the curve given for alloy "E" in Fig. 3 of the paper is the mean of a very large number of results obtained both by ourselves and by Mr. McAllister's group. We can take it that for this pipe that curve is fairly well established.

With regard to pure lead, there is in certain of the pipes which we examined some tendency for small extensions at the slow creep rates, not always traceable to any definite metallographic feature, though appearing generally to correspond to the presence of particularly large grains; but in other sources of pure lead there is in the same material, as far as we have gone (which is round about the range of 0.0001%/hr. average creep rate), no notable tendency to a decrease. As far as we know, there may be some tendency for decreasing extension with increasing grain-size, but it still remains indefinite. That applies also to certain other materials which we have examined. In all cases there is a definite increase in the tendency for intercrystalline cracking to take place with decreasing creep rate, but that is not always associated in any definite way that we can pin down, as for alloy "E" and other materials, with a diminishing extension.

Dr. McKeown said that I had not noted in the paper the extensions obtained on the constant-stress tests. They are not all taken to completion. Some of them were completed as constant-stress tests, and some of them were abandoned as constant-stress tests after 4000 hr. and then left at constant pressure, but I have Tables available which show the results for pure lead and alloy "E" extensions.

Table A shows pure-lead extensions corresponding to the constant-stress tests. The marked results are the extensions obtained at the end of the constant-stress period. In most cases, after about 4000 hr. further pressure adjustments were not carried out. No. 1136 had a constant-stress period carried on for about 10,000 hr. In this case, there was very good agreement with the Andrade curve up to about 6000-7000 hr., and then it deviated from the curve. After that the test was allowed to carry on under constant-

TABLE A.—Constant-Stress Tests—Extension Results. Pure Lead (Screw Press).

Sample Number.	Length, yd.	Hoop Stress, lb./in. ²	Time on Test, hr.	Extension at Break, %.	Extension Remote from Break, %.
2.0 in. outside dia.					
1327	2.5	820	57½	14.3	4.5
1328	2.5	720	645	12.0	5.6
1329	2.5	650	2,190	12.78	6.0
1330	2.5	550	3,942 *	13.3	5.27 *
			10,000		10.0
1332	2.5	460	4,000 *	U	4.0 *
			11,800		6.3
1332	2.5	400	4,000 *	U	2.27 *
			11,800		4.04
3.0 in. outside dia.					
1122	5.0	800	125	11.3	6.43
1128	5.0	800	98	9.0	5.6
1324	2.5	725	516	8.66	8.2
1126	5.0	650	1,416	12.0	7.63
1325	2.5	550	4,000 *	U	4.5 *
			11,200		8.2
1136	5.0	500	10,120	17.33	9.0 *
			13,000		12.0
1326	2.5	450	4,000 *	U	1.0 *
			11,200		2.34
1135	5.0	400	2,100	...	1.3

* Results at end of "constant-stress" period.
U = Unbroken.

pressure conditions; there was an increase in rate of extension over the previous period, but it was not very notable, and, as will be seen, the test went on for about 13,000 hr. The lowest figure, 1.3, in the last column for the last line, is for an uncompleted test.

Table B gives a corresponding set of extension results for the alloy "E" specimens, and the same thing applies here. The actual values obtained as compared with the constant-pressure tests are somewhat difficult to take, because I have had to abandon most of these as constant-stress tests at round about 4000 hr.; but in general we obtain much the same extension value if we plot against the mean rate, dividing the final extension by the total time of test, in a constant-stress test or a constant-pressure test. That, however, is only a rough and ready comparison.

With regard to Dr. Orowan's remarks, and with reference to necking, a very large number of these pipes failed, after long periods, with intercrystalline cracking and no very definite local necking, even in the case of pure lead. I do not believe, furthermore, that there is any very definite ground for thinking that the diminishing extension is a universal tendency. Certainly that tendency exists, and increases with increase of grain-size; but, as Dr. McKeown has pointed out, there is some grain-size at which it seems likely that the same extension will be obtained at any creep rate. I do not know how that fits in with Dr. Orowan's theories.

There is one notion which I have been considering recently. It seems to me that there is a possibility of applying to this type of problem what is known as the equation of continuity. I have no very profound knowledge of that

TABLE B.—Constant-Stress Tests—Extension Results. Alloy "E"
(Screw Press).

Sample Number.	Length, yd.	Hoop Stress, lb./in. ²	Time on Test, hr.	Extension at Break, %.	Extension Remote from Break, %.
3.0 in. outside dia.					
1165	5.0	1535	200	19.35	13.76
1150	5.0	1440	216	27.00	13.20
1154	3.0	1200	912	11.60	10.20
1172	3.0	1000	3,650	10.35	7.50
1174 A	2.5	900	4,224 *	16.60	3.32 *
			4,416		3.50
1174 B	2.5	800	4,200	U	1.33 *
			11,000		2.49
2.0 in. outside dia.					
1333 B	5.0	1420	418	...	12.22
1191	5.0	1200	558	10.25	9.00
1193	5.0	1000	2,706	7.07	5.97
1333 A	2.5	900	3,774 *	3.70	3.23 *
			4,084		3.48
1211	2.5	800	1,643	U	2.54

* Results at end of "constant-stress" period.
U = Unbroken.

matter, but I should like to direct attention to the equation of continuity and give an indication of how I think that it might apply. The equation of continuity is simply that if at any point you have a vector velocity \mathbf{v} (that is, the velocity of a particle in the material that is flowing) the relationship: $\text{div. } \mathbf{v} = -\dot{\rho}/\rho$ applies, where ρ is the density and $\text{div. } \mathbf{v}$ is the divergence of \mathbf{v} . As long as that relationship applies, there is mathematical and, correspondingly, physical continuity of the material, and fracture involves a breakdown in that relationship. $\text{Div. } \mathbf{v}$ is simply the rate of dilation at the point concerned. It can be given by some such expression as $\text{div. } \mathbf{v} = k + m + n$, the values of which are related to the rate-of-strain tensor. Under simple tension, k might correspond to the Andrade κ , and m and n under purely plastic flow might be expected to be of the order of $-\frac{1}{2}k$ each. It seems to me that it is only stating the matter in another way to say that m and n must deviate from $-\frac{1}{2}k$ at some special point, perhaps in a grain boundary, owing either to the initial effects of β flow or to some of these other mechanisms which have been discussed, and this deviation results in a local dilation which carries on as the process continues, and finally results in a breakdown of the equation of continuity and local fracture. It seems to me that applying that sort of relationship to the theories which have been expressed may help us to decide just exactly what is happening in these alloys.

CORRESPONDENCE.

MR. F. R. N. NABARRO,* M.B.E., M.A., B.Sc.: I should like to point out how, by a simple mathematical trick, we can obtain a very sensitive analysis

* Bristol University.

of Dr. Latin's results in terms of the Andrade equation. Dr. Latin gives this equation in the approximate form :

$$\epsilon = \beta t^{1/3} + kt$$

Dividing all through by $t^{1/3}$, we obtain

$$\epsilon t^{-1/3} = \beta + kt^{2/3},$$

a linear relation between $\epsilon t^{-1/3}$ and $t^{2/3}$. Suppose, for example, we apply this method to the top curve of Dr. Latin's Fig. 4 (p. 269). It is clear that Dr.

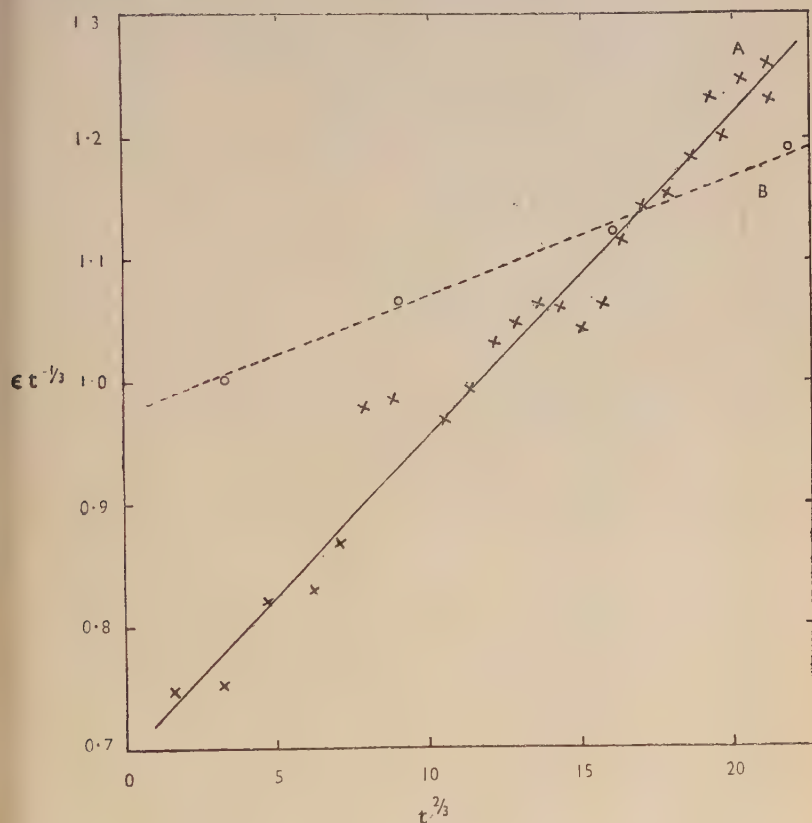


FIG. E.—Pure Lead. Hoop stress = 800 lb./in.² t in hr., ϵ in %.

A. $\beta = 0.6921$, $k = 0.02584$. B. $\beta = 0.974$, $k = 0.00985$.

Latin's dotted curve is quite a good fit; perhaps in the Andrade equation he might have taken a little less β to make the curve rise slower at the beginning, and a little more k to increase the slope at the end. If we read off the experimental values of ϵ (in %) and t (in hours) from the crosses, and plot $\epsilon t^{-1/3}$ against $t^{2/3}$, we obtain the results shown by crosses on Fig. E. These lie very close to the full line, showing that the Andrade formula is a good approximation.

From the computed points on Dr. Latin's curve we obtain the circles on Fig. E, which lie, within the errors of plotting and computing, on the dotted line. To show that the values of β and k corresponding to the full line really do give a better fit than those corresponding to the dotted line, I have plotted in Fig. F the experimental points and the two Andrade curves. The improvement is

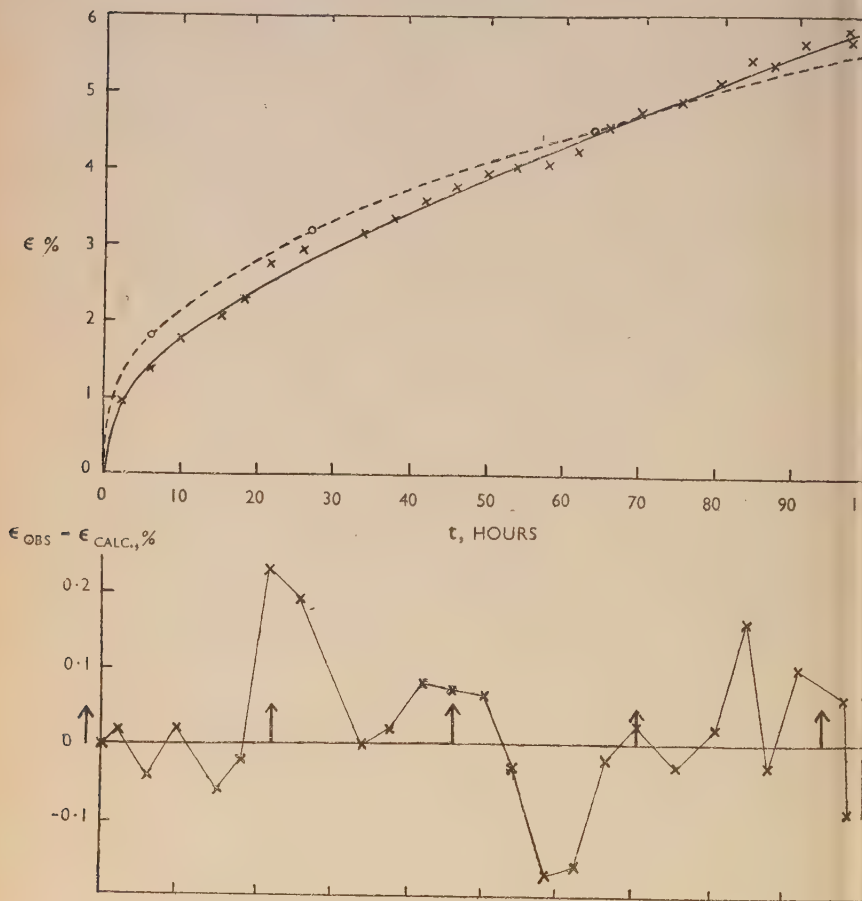


FIG. F.

obvious, and the changes in β and k are large enough to alter the form of Dr. Latin's Fig. 12 (p. 277) appreciably.

Dr. Norman Thompson suggested to me that the deviations from the Andrade curve might be caused by temperature fluctuations, since lead at room temperature is in a range where creep is likely to be very temperature-sensitive. The main variation is likely to be diurnal, and at the bottom of Fig. F the deviations from the Andrade curve are plotted against time. The

arrows are 24 hr. apart, and there is a strong suggestion that the deviations do show the expected 24-hr. periodicity.

Dr. Latin has clearly shown that in alloy "E", grain-boundary cohesion is destroyed more effectively by a given extension by the β mechanism than if the same extension occurs by the k mechanism. I am not sure whether his results justify the view that extension by k creep is entirely innocuous, or even beneficial to the cohesion of the grain boundaries. Perhaps he would comment on this.

A clear theoretical explanation of Dr. Latin's results is not yet available. At least four mechanisms of creep have to be considered, and in polycrystalline lead at room temperature all four are likely to act simultaneously. The first two are connected with dislocations moving across slip planes in the lattice, and will produce rotations of the grains leading towards preferred orientations, with a shearing deformation of each individual grain. We consider that the dislocations are anchored in place by internal stresses; when an external stress is applied, a few of the dislocations are lifted almost to the top of the energy barrier holding them in, and a transient creep occurs as these loosely bound dislocations are lifted over the small remaining barriers by thermal vibrations. At high temperatures, where the metal self-anneals (this includes room temperature for lead), continuous creep can also occur because the potential barriers gradually subside. The other two effects occur at the grain boundaries. In a certain temperature range, the grains in a stressed metal slip over one another until the load is largely carried by those parts of the boundary which are subjected to normal tensions or pressures. This produces a transient creep of very limited extent, always less than the elastic extension. At higher temperatures, the small "keys" which are now carrying the whole load yield by a process which is probably essentially one of recrystallization, and there is an unlimited creep in which the grains rotate in directions depending on their shape and not on their lattice orientations. The grain boundaries are badly deformed, but blocks far from a grain boundary are not distorted. To separate all these effects would require an elaborate series of experiments in which the temperature of test was varied, and the material was observed in both polycrystalline and monocrystalline forms.

The AUTHOR (*in further, written, reply to the discussion and in reply to correspondence*): I should like to express my deep appreciation of the honour of having Professor Andrade open the discussion on my paper, also of his kind and extremely helpful remarks on my efforts.

As regards the technical application of my results, this is, I think, fairly direct. I have dealt with this aspect of the problem briefly in my paper, from which it will be seen that while the conditions in gas pressure cable are such as to aim at preventing any creep in the lead sheath following the initial extension up against the reinforcement, we cannot be absolutely sure that creep is always entirely prevented or that the sheath is stress-free, at least for the first two or three thousand hours. The discovery of the extension characteristics of alloy "E" and materials which behave similarly, seemed to us to be of considerable moment in this connection, and if not positively dangerous in good-quality sheath (i.e. sheath free from extrusion defects) at least desirable to avoid in this special type of application. It was obviously necessary to develop some guiding rules on which we could base our choice of material, &c., for the sheath, and this to my mind clearly involved not merely studies of the "extension/creep rate" relationship but of the whole relationship between the creep process and the fracture process. The conducting of constant-stress tests on pipe, in a similar condition to cable sheath, in an endeavour to analyse the creep process occurring in specimens of this form, with a view to obtaining

if possible an insight into the fracture process, seemed to me one of the best ways of tackling the problem in a reasonable time. I think that I have been successful to the extent of being able to develop a rough working hypothesis which I think I can claim has been of help in arriving at decisions. As regards the general nature of the creep-fracture relationship, I do not pretend to have more than "scratched at the surface" of a problem which is doubtless of some complexity.

I am sorry if I have given the false impression that I consider the third stage of creep a "real feature of flow" under constant stress. I have always accepted the fact that in those cases where this stage is due to stress intensification alone, it is eliminated (assuming also complete homogeneity of material and applied stress) in constant-stress tests. We must also, of course, accept the view that there are other causes of accelerated creep besides pure stress-intensification, and that these may be operative in constant-stress tests.

I am particularly grateful to Professor Andrade for his illustration of the way in which β flow can be conceived of as leading to grain-boundary discontinuities. Whether or not actual voids are produced is a moot point, but I would like to suggest that there is a definite possibility that elastic stresses result. I have in fact followed up this idea by the application of simple vector "equations of dilation" similar to that which I mentioned at the end of the discussion and have arrived at conclusions which I think to be of significance, especially when taken in conjunction with my findings from constant-stress tests on strip, also mentioned at the meeting, namely that the proportion of k to β flow is considerably increased with decrease of grain-size. I cannot deal with these results in detail here, but the general picture is that of local elastic dilations at grain boundaries, the extent of which depends, among other factors, on the relative values of β and k .

I should like also to express my gratitude for Dr. Orowan's extremely interesting contribution. His analysis of ductile fracture in creep is of particular interest, but I think he will agree that it is doubtful how far it can be applied to the case considered here, which is chiefly concerned with fracture of essentially brittle type. It would appear also at first sight to be somewhat at variance with our results on the fine-grained alloys in which ductile fracture occurs, and there is apparently an increase of general extension with decreased "steady" creep rate.

These results, however, were obtained under constant-load conditions and not constant stress, and there are probably other factors (e.g. recrystallization) coming in. Ductile fracture is of less direct importance to us than the intercrystalline type in the applications with which we are concerned, but we have been carrying out a fairly detailed investigation into various factors which influence the extension obtained (at fairly high creep rates) when this type of fracture occurs, and some very interesting results have been obtained, which cannot, however, be detailed here.

Dr. McKeown has mentioned several important points, with some of which I have already dealt. I have given some consideration in my paper to the difference in stress system involved in pipe tests and strip tests and think that I can say further that the general effect as compared with unidirectional stress is to diminish the proportion of k flow, a fact which may be of some significance. I think it of interest to mention some tests in which a state of bi-tension was deliberately applied. These are tests which I have developed on discs cut from pipe or sheet, subjected to pressure at one side so that a bulge forms. These tests have resulted in an enhanced liability to intercrystalline fracture and have given us some valuable information, to which I have briefly referred in my paper. I am in full agreement with Dr. McKeown's findings with regard to the effect of grain-size on the initiation of stage-3 creep, but do not think

that they affect my comparison of pure lead and alloy "E", in the states considered, in any way.

I should again like to assure Dr. Chaston that the extension curve given for alloy "E" is very well established (again for pipe *in the state considered*). The individual results on the screw-press pipe are shown in Fig. G. Very similar results were obtained for ram-press pipe of similar quality. Qualitatively similar results were also obtained on strip samples taken longitudinally from the pipe and on directly extruded strip of similar grain-size. I can also emphatically assure Dr. Chaston that the failures were not on seams.

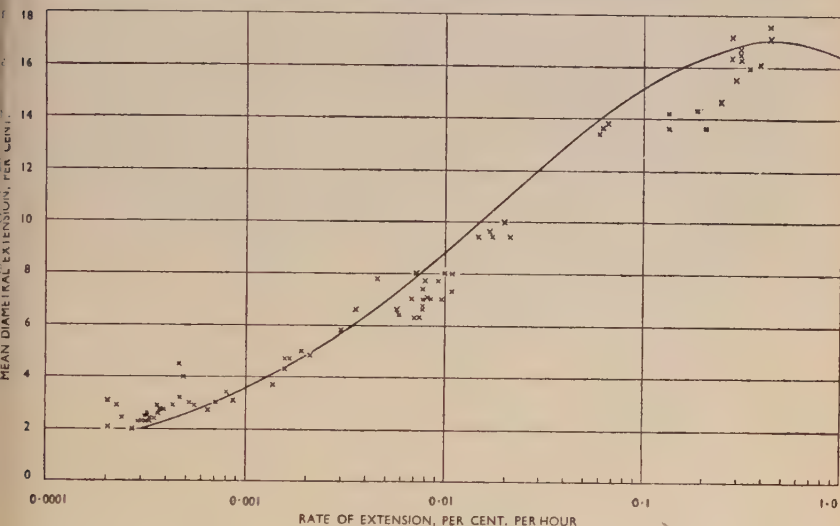


FIG. G.—Individual Extension Results on Alloy "E" Pipe.

I think that Dr. Chaston is under some misapprehension as to the exact purpose of this work. I have not pretended that it should be a purist physico-metallurgical investigation, but a piece of industrial research fitted into a comprehensive range of more conventional tests and aimed at supplementing the information from these tests to give us increased confidence in our decisions on an urgent problem. In this I consider I have been successful. I do not feel that I need apologize in any way for the experimental technique, the tests being carried out meticulously in the best conditions possible at the time. The investigation has naturally developed to a certain degree into a metallurgical investigation in its own right, so to speak, and considerable effort is being expended in continually improving technique.

I thank Mr. Hopkin for his interesting and useful observations, and I have studied with great interest reports on his own work on this subject, under Dr. McKeown. We are, of course, fully aware of the heterogeneous nature of extruded pipe (it is worth noting, however, that the structure is symmetrical), but I would point out again that what I was aiming at was an effort to obtain values of the constants more or less representative of actual cable sheath.

Mr. McAllister's group has also carried out extremely comprehensive and detailed investigations on these pipes, sheaths, strips, &c. (many of his results

on alloy "E" pipe are included in Fig. G), and it is pleasing to record that we have achieved a large measure of agreement in our results, conclusions, and views on these matters.

I think that Mr. Nabarro's valuable contribution should go far towards clarifying matters, and I was very pleased indeed to receive it. Dr. Thompson is right in ascribing the fluctuations to temperature variations; these were unavoidable, but I think by no means so serious as greatly to affect the general conclusions. A maximum variation of about $\pm 2^\circ$ C. was usually obtained. Painstaking efforts are being made to improve matters in our further investigations.

Mr. Nabarro is in error in thinking that I have regarded k flow as being necessarily innocuous. If it is not prevented it most obviously is not innocuous, and in practice all our efforts are bent on preventing its occurrence in pressure cable. In point of fact, I consider that the relative values of β and k are more important from several points of view than their absolute values, but that in any case, particularly for very slow creep, both contribute to the observed effects. The general picture which I have in mind is that of grain-boundary regions which are caused by flow restrictions of one kind or another to dilate elastically by the surrounding plastic or visco-plastic extension, to which both β and k flow contribute. k flow has, however, "self-recovering" properties which increase with the value of k .

DISCUSSION ON PAPER BY MR. N. DUDZINSKI,
MISS J. R. MURRAY, MR. B. W. MOTT,
AND DR. B. CHALMERS : "THE YOUNG'S
MODULUS OF SOME ALUMINIUM AL-
LOYS." *

(*J. Inst. Metals*, this volume, p. 291.)

PROFESSOR L. AITCHISON,† D.Met., M.Sc. (Member of Council) : I am very glad to have the opportunity of saying something about this exceedingly interesting paper. I am sure it is unnecessary for us to compliment the authors on or to criticize the actual work; we expect work of a very high quality to come from the laboratory in question, and this paper is no disappointment in that regard. I believe it is quite likely that in years to come, when people look back on the development of metallurgy in this country, they will quote this paper as the beginning of a new epoch, because it takes us as an Institute and as an industry into a field which has been singularly neglected. Possibly the authors timed their paper very well, so that we could have the "new look" in metallurgy contemporaneously with the "new look" in other spheres of influence.

We are reminded by the authors that in the past it has been generally believed that the modulus of elasticity of an alloy was approximately calculable from the previously determined values of the constituent elements, taking into account the chemical composition of the material; but it is clearly shown by the present workers and by other theoreticians that we cannot arrive at a correct value of Young's modulus in that way, and that in place of the elements which compose the alloy we must substitute two other things: the matrix and the compounds which are likely to be produced within the material. That, I think, is exceedingly important, because up to the present I believe that intermetallic compounds have not been particularly popular in non-ferrous structural materials. We have a passing affection for those compounds which will pop in and out of solution and therefore make heat-treatment both profitable and fascinating; but we have not quite reached the stage of accepting intermetallic compounds in the same way that our ferrous brethren do, because they have been brought up on iron carbide from their cradles.

I think that this enhanced interest in intermetallic compounds is all to the good, and I hope it may be possible for the theoretical work mentioned in the paper, which has been done by Mr. Middleton and others to show the basis of calculation, to be made more generally available. I do not think that I am giving away any secrets if I say that Mr. Middleton's approach is roughly that of taking the Southwell hole in a plate and filling it with another phase; so that it is possible to achieve a straightforward mathematical consideration of these materials, based on the conception that they consist of a clamped-up aggregation of perforated plates. The engineer in the past has made many calculations on perforated plates, and is accustomed to deal with two-phase materials, one being metal and the other being

* Discussion at the Annual General Meeting, London, 17 March 1948.

† Professor of Industrial Metallurgy, University of Birmingham.

wind. Now, stimulated by the present authors, he can replace wind by another metallic phase. That is a most interesting and valuable contribution.

Clearly, if the theoretical results obtained in this way are to be proved, it is desirable that we shall know something fairly specific about the properties of those compounds which may be employed in preparing alloys with a high modulus of elasticity. I believe work is going on in a good many places, and Dr. Raynor may refer to some that he is doing on the isolation of these compounds. He is probably going about with a few pieces of inter-metallic compound in his pocket, of the size of a silver threepenny-bit. I do not think he carries them round to cheat the Revenue by putting them into stamp machines; he does so in the hope that somebody will tell him the best way of determining the moduli of these crystals in the direction of their various crystallographic axes. He may be able to tell us something about that work later on.

The authors may not have done full justice to the mechanical properties of these alloys. They have suggested that it is possible to obtain alloys with an increased modulus only by the sacrifice of other mechanical properties. That may or may not be true, but I should like them, if they can, to deny the full implication, because it is possible it could be carried too far. In any case, the properties described in this paper are sufficiently attractive to aircraft structural engineers, provided a reasonable increase in the modulus of elasticity can be obtained. Some doubts on this point were expressed at an early stage, but the matter has been carefully sifted by investigations carried out independently by Professor Pugsley and Mr. H. L. Cox, both of whom have confirmed that on the figures given here for modulus and proof stress the alloys are such as would result in weight-saving in aircraft structures, which, of course, is supremely important.

That leads me to the point from which I started, that metallurgy now must take full notice of this development. It seems to me that this paper calls for very active co-operation by industrial manufacturers of alloys, so that the ideas and concepts whereby our metals may be built up from their constitution and not just taken as they come, may be carried further and put to practical use, particularly for structural-engineering purposes.

I have only one question to ask. I think that I know the answer, but should like it confirmed. The engineer is particularly anxious to know whether the modulus of rigidity goes with the modulus of elasticity, as he attaches considerable importance to the modulus of rigidity. Can the authors assure us that it may be assumed that the rigidity modulus goes in parallel with the elastic modulus?

I congratulate the authors very heartily on a most valuable and interesting paper.

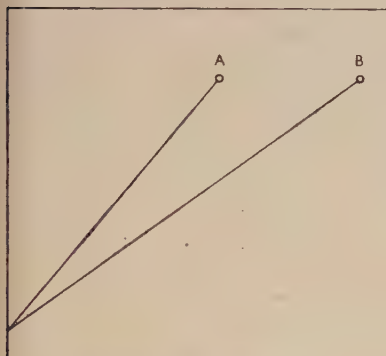
DR. G. V. RAYNOR,* M.A. (Member): In this most valuable paper, it is shown that alloying aluminium with several elements—manganese, cobalt, beryllium, nickel, and silicon—significantly raises the value of Young's modulus by amounts proportional to the concentration in weight percentages. The effectiveness of the solute apparently depends firstly on the value of Young's modulus for the phase which is associated with the aluminium-rich matrix and secondly on the amount of this phase which is present. The question now arises as to whether there are other solutes, in addition to those studied, which could be used, perhaps more effectively, for the same purpose.

The Young's modulus of beryllium is one of the highest known for a pure metal, so that it is most unlikely that any significant improvement

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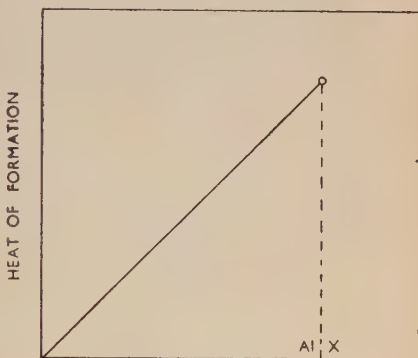
over the values already obtained by the authors could be obtained by the addition of any pure metal which does not form intermetallic compounds with aluminium. Intermetallic compounds must therefore be relied on to give any possible improvement.

We can see that, if we have to choose between two compounds of equally high Young's modulus, the increase in Young's modulus in the aluminium-rich alloys per unit increase in composition is going to be greater for the compound containing the most aluminium. Assuming that the effect is strictly additive, then plotting the Young's modulus against composition for two compounds *A* and *B* of equal Young's modulus, the slope of the curve for the compound containing the more aluminium is clearly greater than that for the other (Fig. A).



COMPOSITION, PER CENT SOLUTE

FIG. A.



Al

COMPOSITION, ATOMIC PER CENT SOLUTE

FIG. B.

The other factor is the Young's modulus of the compound, which should apparently be as high as possible. Unfortunately, there are very few data indeed, for the Young's modulus of intermetallic compounds, which can be used as a basis of choice. I want to suggest, in agreement with what the authors write on p. 309, that the heat of formation of an intermetallic compound may be taken as a measure of Young's modulus, for the purposes of selection. As far as I am aware, there exists no rigorous proof of this relationship; I think that it has been taken as self-evident. Qualitatively speaking, we can see that the higher the heat of formation, the tighter will be the binding between atoms, and therefore the higher the Young's modulus may be expected to be. Let us consider a compound AlX , where X is a solute which forms only very limited solid solutions in aluminium. If we plot heat of formation vertically against the atomic composition horizontally, the graph of the heats of formation of the alloys will be a straight line (Fig. B). What I want to suggest now is that Figs. A and B can be considered as analogous.

The heats of formation of some of the compounds in which we are interested in this connection, namely $NiAl_3$, Co_2Al_9 , and $FeAl_3$, are respectively 9.5, 7.7, and 6.1 kg.cal./g.-atom. If these values are plotted as in Fig. B, the result is as shown in Fig. C. The curve for cobalt lies above that for nickel, in spite of the fact that Co_2Al_9 has a lower heat of formation than $NiAl_3$, and the effect on the aluminium-rich alloys produced by cobalt would be expected to be somewhat better than that produced by nickel, which is what

the authors find. The heat of formation of FeAl_3 is lower than that of NiAl_3 , and there is the same amount of aluminium in the compound; iron should thus have a smaller effect than nickel. The heat of formation of MnAl_6 is not known, but can hardly be lower than that for FeAl_3 ; manganese should thus be more effective than cobalt, which is, in fact, the case. With chromium the heat of formation is again not known, but, assuming that the heat of formation of CrAl_7 is of the same order as that of MnAl_6 , chromium would be expected to be a little more effective than manganese. As far as copper is concerned, CuAl_2 has the very low heat of formation of 3.2 kg.cal./g.-atom, and copper would not be expected to be at all effective in raising the Young's modulus of aluminium. Proceeding in this way, it should be possible to assess whether or not a given solute is likely to form an effective addition to aluminium for the purpose of increasing the Young's modulus of the aluminium-rich alloys.

Fig. C has been drawn in atomic percentages. For strict comparison with the diagrams in the paper, conversion to weight percentages is necessary,

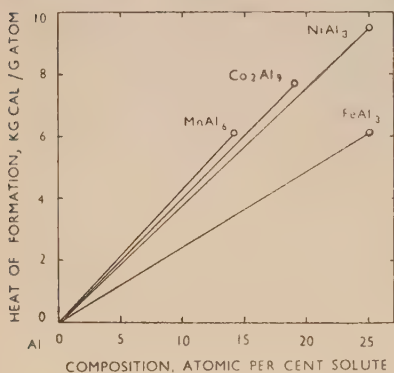


FIG. C.

but this, apart from introducing a slight curvature, does not alter the expected order of effectiveness.

There is a further possibility. It is very speculative, but it may just be worth mentioning. The heats of formation of certain metallic nitrides are very high indeed; the heat of formation of titanium nitride is 40.2 kg.cal./g.-atom, which may be compared with the values for the intermetallic phases discussed. It might be possible to allow an aluminium-titanium alloy at a suitable temperature to react with gaseous nitrogen and form titanium nitride, and thus to develop an aluminium-rich alloy with quite a small amount of alloying constituent, but containing

hard particles with very high Young's modulus to give the increased effect for the aluminium-rich alloys. If such means failed, there is always the possibility of introducing any hard particle artificially by the methods of powder metallurgy.

I think that Professor Aitchison has said almost all that I would wish to say about the intermetallic compounds which we are preparing, but I would mention briefly that we have been interested for a long time in how intermetallic compounds behave, and particularly to what extent they dissolve other metals. To investigate that, we have been separating from aluminium-rich melts pure samples of many of the compounds about which we have been talking to-day. The way in which it is done is quite simple and not new in principle, but it happens to work very well for these alloys. We cool a small ingot very slowly, the composition of the ingot being such that the compound in which we are interested comes out as primary crystals which segregate at the bottom on slow cooling and grow quite large. The ingot is then made the anode in a suitable electrolyte. For many of these compounds, normal hydrochloric acid will do. On electrolysis with a suitable current density, the aluminium-rich matrix dissolves away and the compound crystals drop to the bottom of the beaker and can be collected, washed,

dried, and in our case analysed and the X-ray diffraction pattern taken. We are dependent on the growth characteristics of the crystal for the shape of crystal which we get by this method. We may get needles or plates, and the crystals of which Professor Aitchison spoke were prepared from an aluminium-iron-silicon alloy. We can also separate, from the same alloys within a different composition range, small polygonal-shaped crystals. Those are the three shapes which are available at the moment, for which it might be possible to determine the Young's modulus. If anybody can advise us with regard to a suitable experimental method for use on such specimens we shall be most grateful.

DR. H. SUTTON* (Member): As the authors observe in the introduction, an effective increase in the Young's modulus of high-strength aluminium alloys would be of great value for aircraft designers. Their experimental work constitutes an intriguing approach to the problem of alloys having a higher modulus. One interesting feature of the work is the apparent insensitivity of their alloys to condition as regards cast condition compared with wrought, or as cast compared with heat-treated, which may not be the case with alloys showing greater solid solubility. In the early days of beryllium bronze, as Professor Aitchison and others may remember, a good many of us in this country were interested in some results published by Masing and Haase.† These authors measured the rigidity modulus of samples which had been solution treated at about 800° C., a normal temperature for beryllium bronze in those days, and they carried out similar determinations of modulus of rigidity on samples which had been age-hardened at various temperatures. They observed very substantial changes in modulus up to about 29%, and the increase in modulus went on with progressive rise in the age-hardening temperature up to, I think, about 400° C., and after that the modulus began to drop again. There was something unusual about the age-hardening treatments, which I think were applied consecutively, but those experiments did bring to light the important influence, in that series of alloys, of the precipitated phase.

The curves of hardness resulting from this precipitation treatment were rather interesting and indicated that the hardness peak occurred not very far away from the maximum value for modulus of rigidity. The work was repeated in this country, and similar increases in the values of rigidity modulus were noted, but on the material used the increase was not quite so great. Somewhat lower values, for samples of a fairly similar material, were obtained, but an increase of modulus of over 20% was observed. The temperatures recorded in the British work corresponded very closely to those in the German work.

Guillet‡ has called attention to the importance of the heat-treatment effects where there is solid solubility of a component, and to the varying influence of compounds of different types. He has referred to copper alloys containing, for instance, 13% aluminium and 25% tin, in which the annealed alloys have Young's modulus values some 60% higher than the quenched alloys. These results suggest that where there is a considerable solid solubility of the constituents, the effects of heat-treatment may be very great.

Templin and Paul§ some years ago made some very interesting observa-

* Ministry of Supply.

† G. Masing and C. Haase, *Wiss. Veröff. Siemens-Konzern*, 1920, 8, (1), 142.

‡ L. Guillet, *Rev. Mét.*, 1939, 36, 497.

§ R. L. Templin and D. A. Paul, *Trans. Amer. Inst. Min. Met. Eng., Inst. Metals Div.*, 1931, 480.

tions on the effect of heat-treatment on cast aluminium alloys containing magnesium and also on cast aluminium alloys containing nickel and copper and one or two other elements. They found that an increase in the modulus of 25% or so could be obtained in aluminium alloys containing about 10% copper or 7% nickel. They also noted that when the magnesium constituent was taken into solution in cast aluminium-magnesium alloys, the modulus was reduced.

I hope that the present authors will be able to continue their work, and that others will feel stimulated by the publication of the paper to follow similar lines of study. The subject is, in my view, a most important one from the scientific point of view, and also abounds in practical interest and importance.

MR. L. ROTHERHAM,* M.Sc. : It is not easy to follow the three previous speakers, because each of them has referred to something that I wanted to say. In any case I am a little restricted in the comments which I can make, because this work is still continuing, and since I am now responsible for its progress, it would be a little unwise on my part to take an over-critical view of the paper. In any event, I do not want to say anything very critical, but rather to endorse the remarks which Professor Aitchison has made and to point out the fact, to which Dr. Sutton referred at the end of his remarks, that this study is a particularly satisfying one, combining a wide variety of interests. First of all there is the scientific problem, secondly an interesting metallurgical development investigation, and finally there will be the straightforward engineering proposition of applying the alloys in aircraft structures.

This paper, as I read it at any rate, is an outline of the introduction to the scientific side of the work, but reference is made to the beginning of the development stage of the programme and to the further work which is needed to clear up some of the unsettled points in different alloy systems. It is fair to point out that the authors have worked out some of the easier systems, and have left to those of us who follow some of the more difficult ones; but I think that they were very wise to make such a choice.

On the development side, there is a reference in the paper to the work which Stalker has been doing on more complex alloys, and here quite substantial progress has been made, though there were some initial difficulties in the heat-treatment of some of the alloys. This development work has been extended by one of the authors (Mr. Dudzinski), and I think it will probably be of interest to say that the final properties which have been obtained coincide almost identically with those properties which are listed in this paper (p. 308). I mention that not only as a matter of interest for its own sake, but as a demonstration of the very sound way in which the foundations of the study have been laid by the authors of the present paper.

Most of the remainder of what I wished to say has been covered adequately by Dr. Raynor, but there is just one point which arises in connection with the diagram which he drew, and that is that the modulus of these different intermetallic compounds differs only slightly from compound to compound. In actual fact, if you extrapolate the curves to 100% intermetallic compound a rough value of modulus can be obtained for the compounds and you will find that they are not very different one from another. Manganese has been demonstrated to be the most effective addition element, and from what has just been said this is not because manganese itself is particularly effective, but because it produces a lot of compound. That is a point which Dr. Raynor mentioned, but perhaps did not emphasize sufficiently. The objectionable feature of this conclusion is that I think that it is going to make the situation

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rather difficult when we come to the combination of properties which will be wanted in engineering, the combination of high strength and high modulus; if we have to have a large quantity of a brittle constituent in our heat-treated alloy, the authors' prediction that we shall have to sacrifice something in ductility and proof stress is a true forecast. Dr. Raynor has also pointed to the way out, however, and that is the use of compounds with very high heats of formation, where we may hope to get a substantial increase in the over-all modulus for only slight additions of the intermetallic compound.

MR. A. J. MURPHY,* M.Sc. (Vice-President): I agree very strongly with what Professor Aitchison has said about the importance of this paper as marking a new development. Up to the present the exploration has been to confirm the additive nature of the Young's modulus, and to find how far we can go by enriching the alloys in high-modulus elements or compounds before causing excessive deterioration of the other desirable mechanical properties. At present elongation seems generally to be the limiting factor. If we can find in complex alloys that from a given addition of element, by suitable manipulation—as, for instance, heat-treatment, mentioned by Dr. Sutton—we can obtain more than a proportional effect on the modulus, interesting possibilities will be opened up.

I should like to emphasize one other point, as illustrating the limitations which may affect the application of these alloys. Materials of high modulus would be most valuable, as mentioned in the introduction, in slender structural members which are liable to be put into compression. Such parts are made of rolled sheet and rather fine extrusions; and that means that the working properties of these materials are also going to be rather critical. Castings are not involved generally in these applications, and for alloys which may only be forged into massive sections there is not much interest; but it will be necessary for the technical exploitation of these high-modulus materials that they shall be capable of being rolled and extruded into relatively thin sections.

MR. D. McLEAN,† B.Sc. (Member): There are two points which I should like to raise. The first relates to Fig. 1 (p. 297). If one extrapolates the various curves back to zero %, they give different points. Am I correct in assuming that that is due to a solid-solution effect, and, if so, have the authors any idea of the principles governing the effect?

Secondly, if the general treatment put forward here is correct, it should apply to other systems, and in particular it should apply to ferrous systems, which would, of course, give the possibility of making alloys with very high Young's moduli indeed. The heat of formation of the carbides found in steels varies widely, but according to the International Critical Tables the heat of formation of Cr_3C_2 is 136 kg.cal. per gram formula weight. Even allowing for the fact that iron starts off with a Young's modulus of about 28 million lb./in.², that should be enough, by comparison with the figures given in this paper, to make a significant improvement in the Young's modulus, particularly as it is possible to control to a large extent the distribution of the carbides, a factor which presumably has some bearing on the problem. However, in practice it is found that the Young's modulus of steels is always between 28 and 32 million lb./in.², whatever the composition and heat-treatment. I should be interested to know whether the authors have any observations to make on that point.

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† Metallurgy Division, National Physical Laboratory, Teddington.

MR. J. W. JONES,* M.Sc. (Member): There is very little that I can add to what has already been said. Professor Aitchison has picked out the salient point in the discussion immediately with his apt phrase about the "new look". On reading this paper, I at once went to one of my colleagues who is a design expert in aircraft and asked him which he would prefer: an alloy with a proof stress figure of 21, an ultimate of 28, an elongation of 4, and a figure for E of 12, or his usual alloy. He at once said, "Oh, I do not know; with an E of 12 I should have to begin to think afresh about the whole problem". This idea will not produce immediate results; it will produce a set of problems for technical men, who in my experience are a trifle conservative.

There are one or two points to which I would direct attention with regard to these properties in aircraft structures. First let me talk about bend stress. What is the justification for these new higher- E alloys? In the case of an aircraft structure, it must not bend, or at least it must bend as little as possible. Even elastically it must not bend, because the moment the shape changes it has an effect. The aircraft is designed for performance on its shape and on the relative position of wings and tail and so on, so that as soon as the structure begins to bend it begins to lose its aerodynamic efficiency. There is therefore a strong case for a stiffer material, a material which under a given stress will bend less.†

In ordinary practice, of course, the aircraft-structure designer has always taken care of this quite well by taking full advantage of the moment of inertia. By the usual arrangement of what to this audience I will call girders and supports, the structure as a whole has been made stiff and strong, but increasingly aerodynamicists are looking forward to more efficient structures,

* Senior Lecturer in Materials and Metallurgy, College of Aeronautics, Cranfield.

† Since making these remarks, a colleague of mine, Mr. W. S. Hemp, M.A., has kindly supplied the following figures, illustrating the usefulness of these alloys for lower loadings, and conditions above which there is no advantage in the saving of weight by the use of an alloy with these higher values of E but also increased density. He also confirms the desirability of increased proof stress, if advantage is to be taken of increased E .

Maximum compressive stresses for stringer-skin combinations:

$$f = 0.9 \sqrt{\frac{EP}{l}}$$

P = load per in. (compression)

l = rib pitch

Take $E = 10 \times 10^6$ $p_1 = 21 \text{ tons/in.}^2 = 47,000 \text{ lb./in.}^2$

$P/l = 50, 100, 150, 200, 250, 300$

$10^{-6}EP/l = 500, 1000, 1500, 2000, 2500, 3000$

$10^{-3}\sqrt{EP/l} = 22.4, 31.6, 38.8, 44.7, 50, 54.9$

$f = 20,200, 28,400, 34,900, 40,200, 45,000, 49,400$

With $E = 12 \times 10^6$:

$f = 22,200, 31,100, 38,200, 44,100, 49,400, 54,100$

With $E = 14 \times 10^6$:

$f = 24,000, 33,600, 41,200, 47,600, 53,400, 58,500$

- (1) Appreciable weight saving can be achieved for the lower loadings $P/l \leq 150$. For the values of E considered (neglecting variation of density) we have weights in the ratios: 1 : 0.91 : 0.85.
- (2) For higher loadings we soon meet the proof stress and so no advantage is obtained. At $P/l = 200$ we might use $E = 12 \times 10^6$, but $E = 14 \times 10^6$ would not save weight. For $P/l \geq 250$ there is no advantage.
- (3) The argument of (2) can be met by increases in proof stress. A p_1 of 28 tons/in.² = 62,600 lb./in.² would make even materials with $E = 14 \times 10^6$ economical right up to $P/l = 300$.

so as to have much thinner wing structures. Here the geometry of design will not allow for the use of these structural methods of giving stiffness, and so an increasing demand will be made for a material which is by nature stiff without being reinforced. Particularly would this happen on thin leading edges, so that the metallurgist is not getting going too soon to produce for the aircraft engineer some better alloys.

I do not feel altogether happy, however, about the way in which this paper suggests that progress is being made. In the first place, we heard this morning that there is to be more latitude allowed for giving diagrams in papers, and I wish that we could have had drawn for us some typical stress-strain curves for these materials. The usual sort of alloy with which we are associated gives a curve from which we take off the proof stress on which the engineer designs. I hope that the authors will correct me if my deduction is wrong, but it seems to me that they are producing a type of alloy with a stress-strain curve such as is shown in Fig. D. The behaviour of the material in the looped part of the diagram is of the first interest to the aircraft designer, and I could wish that the paper had given us more information about that.

The point has been made by other speakers that there is a limited attraction in alloys which are less strong but have a higher E , because if they are less strong the question of break stress as well as bend stress arises. We must design the aircraft so that it does not break, and if in order to get the necessary break strength the material must be thicker, because the material has a lower proof stress, then in any case we should get the stiffness with the thicker material, and I cannot see that there is any particular advantage accruing to us with a material which has a higher E but a lower proof stress.

I notice that the projected alloy which is going to be made is an alloy of nickel, copper, and so on. I will illustrate the point that I want to make by reference to the alloys in Fig. 2 (p. 301). The attractive alloying element in Fig. 2 is beryllium. Here the next essential point comes in, that of weight. Our new alloy must not be heavier than the existing alloys to obtain the same stiffness and the same strength. If we alloy with beryllium, my rough calculation is that the typical sort of specific gravity of these aircraft alloys is about 2.65-2.70, and if we alloy with manganese, on the basis of 10% the specific gravity figure goes up to 3.1, whereas with beryllium it will remain in the usual region of about 2.65. That may not seem a great deal, but it would mean a great deal on an aircraft structure. The lb./in.² of the beryllium alloy, 0.095, rises with the manganese alloy to 0.11. To make an alloy with the necessary mechanical properties but with increased weight will, I feel, seriously decrease its attractiveness to the aircraft industry. I should like to feel, therefore, that more attention was being paid in future to the 10%-

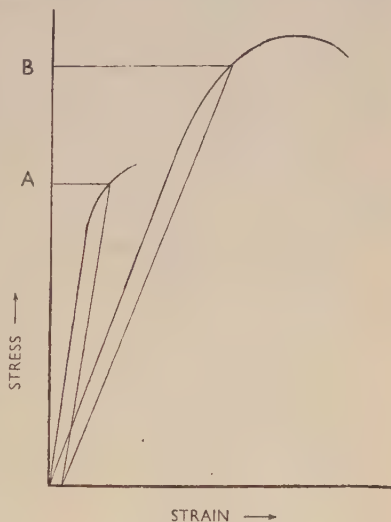


FIG. D.—A has greater E but smaller proof stress than B.

silicon alloy alloyed with beryllium, rather than to the heavier alloys alloyed with manganese, copper, and nickel.

The PRESIDENT (Sir Arthur Smout): I should like to congratulate Miss Murray on her maiden effort and on the very concise manner in which she presented the paper, thus giving us ample time for discussion. I would remind her that there are at least two distinguished ladies who have left their mark on this Institute. I hope that she will follow in their tradition, and that we shall hear a great deal more of her in future. It is most encouraging to think that we have Student Members in this Institute who are capable of playing their part in preparing and presenting a paper of such high calibre as this.

MR. N. DUDZINSKI (*in reply*): I have not much to say regarding Professor Aitchison's contribution to the discussion, but I should like to remark that the modulus of rigidity of alloys should change with composition similarly to the Young's modulus.

With regard to the excellent review of the problem given us by Dr. Raynor, I should like to refer to results which I obtained recently on some aluminium alloys which might throw more light on this problem. I have been investigating the elastic properties of some aluminium-rich systems on the lines suggested in the paper and also put forward in a private communication by Dr. Raynor, and I list them in the order of their usefulness:

Alloy.	Increment in E per 1 wt.-% of Added Element, $E \times 10^{-6}$ lb./in. ²
Aluminium-calcium	-0.33
Aluminium-magnesium	-0.08
Aluminium-copper	+0.08
*Aluminium-tungsten	+0.10
*Aluminium-zirconium	+0.10
*Aluminium-columbium	+0.15
*Aluminium-molybdenum	+0.21
Aluminium-iron	+0.23
*Aluminium-boron	+0.28
*Aluminium-titanium	+0.295
Aluminium-vanadium	+0.325
Aluminium-chromium	+0.39
Aluminium-manganese-chromium (with the ratio of Mn to Cr 4 : 1, annealed at 550° C. for prolonged time and cold-water quenched, having a ternary phase "G", $(\text{MnCr})\text{Al}_{13}$	+0.52

In systems marked with an asterisk the figures are not final and might be subject to revision as only a few sound samples were obtained. If we compare these figures with the values of the heat of formation of inter-metallic compounds which come or are likely to come into equilibrium with " α " solid solution, we see that calcium addition causes a drop in E in spite of the high heat of formation of CaAl_3 compound. Of course this might be due to a very low Young's modulus of metallic calcium. I have examined the Young's modulus of calcium and found it to be of the order of $2.8\text{--}3.2 \times 10^6$ lb./in.²; the American workers obtained an approximate figure of 3.5×10^6 .

Judging by the heat of formation of FeAl_3 compound, the increment in

E should be lower in Al-Fe alloys than in Al-Co and Al-Ni alloys, which is in disagreement with experimental results. Alloys Al-Cu, Al-Cr, and Al-Ti show the increments in E which follow the rule of the heat of formation. Some systems quoted above are difficult to comment on as they involve peritectic reactions and the heat of formation of intermetallic compounds formed in them are not known.

I tried to correlate the E of aluminium alloys with the place in the Periodic Table which is occupied by the alloying elements concerned. If we plot the increment in Young's modulus per 1 wt.-% of alloying element against the atomic number of these elements we observe then an increasingly beneficial effect on E from Be to B, from Mg to Si, from Ca, through Ti, V, to Cr. Then a steady decrease takes place from Cr, through Mn, Fe, Co, Ni, to Cu. In the higher atomic number range, ΔE increases from Zr through Cb to Mo. In each group of the Periodic Table the element lying below shows a lower ΔE compared with the elements lying above, i.e. Be > Mg > Ca, Ti > Zr, V > Cb, Cr > Mo > W. Silicon is an exception to this rule, probably owing to the fact that it does not form any intermetallic compounds with aluminium. This suggestion is rather speculative and the problem is open to discussion, as only a few alloying elements were investigated in aluminium alloys, and some results might have to be revised if more experimental data are available.

There is also another question of whether the electron-concentration rule could be applied to the intermetallic compounds of the transition elements. If such is the case, although it might seem to be highly speculative, a suggestion would be put forward that the compounds with higher ratios of electron concentration to atomic number of alloying element might cause the higher ΔE in some aluminium alloys.

Dr. Sutton put forward a suggestion that the heat-treatment of the copper-beryllium alloys proves beneficial in raising the E . It was proved by French workers that by an addition of up to 15% of beryllium to copper, and by heat-treatment, a considerable increase in elastic properties was obtained, and the Young's modulus of that alloy was as high as that of steel. I did not understand whether Dr. Sutton meant that by quenching copper-aluminium alloys a higher or a lower modulus was obtained.

DR. SUTTON : A lower modulus.

MR. DUDZINSKI : It is apparently true that in those alloys in the region of eutectoid composition, quenching has a marked effect on the Young's modulus, decreasing it considerably; on annealing, the E recovers its former value. Quenching of the alloys of the composition corresponding to that of a homopolar compound, e.g. Cu_3Al_4 , might lead to discontinuities such as cracking, owing to the poor heat conductivity of the compound. If this were the case, E might not recover its former values after annealing.

Mr. McLean suggested the application of the idea of the heat of formation to the iron-carbon system. Unfortunately, not much is known about the variation of the Young's modulus of steel with the percentage of carbon content; not much research has been done on this subject as at one time it was believed that those properties were constant. I should say that very little change in E with carbon content is to be expected in carbon steels. The heat-treatment of carbon steels involves also slight change in E . Rather a higher E is to be expected in some alloy steels such as those with tungsten. This could be ascribed to the formation of a tungsten carbide of homopolar bonding and of a high heat of formation. It has been proved by some workers, especially in France, that homopolar compounds give a higher E . They record an increase of up to 40% compared with the compounds of metallic bond.

Mr. Jones is interested in the question of whether aluminium-beryllium alloys could be used, perhaps with 10% silicon. Although the density of such an alloy would be favourable, we do not expect much from that system, because silicon does not form any compounds with aluminium, and beryllium does not form any intermetallic compounds with aluminium either, and therefore we do not expect any heat-treatment effect, and so on. Some time ago a good deal of hope was felt about beryllium in America, but there is not so much now.

CORRESPONDENCE.

DR. F. A. FOX * (Member): This paper is to be welcomed as the first publication on an important approach to a topic of fundamental engineering importance. It seems certain now that this paper has appeared that work will be stimulated in many places, to follow up the authors' ideas in their application to many materials. The work is, perhaps, of greatest importance in its application to critical aircraft structures, and a comparison with materials of "sandwich" construction naturally comes to mind. There seems to be no reason why parts subjected to bending should not be so fabricated that the modulus of elasticity will increase as the distance from the neutral axis increases. In other words, it might be possible to make highly stressed parts with concentrations of critical second phase which progressively increase towards the outer layers of the piece; to achieve such results powder-metallurgy methods would probably be essential. It might also be that a similar idea could be applied in other ways to make the distribution of second phase coincide with the distribution of stressing. For example, the second phase might be so distributed as to give a fibrous texture, taking on a desired pattern which might be found to give the best results under stress.

In some pure metals the modulus of elasticity varies very considerably with the crystallographic direction, the variations sometimes being of the order of, say, 5:1. A study of the directional effects of the modulus of elasticity of the second phase may, therefore, be of considerable importance, although it is on general grounds to be expected that such large variations of modulus with direction are not to be expected in intermetallic compounds of the kind likely to be important in this field. Nevertheless, should it prove that in some systems directional effects are important, then it would be indicated that the second phase should be produced in the body of the metal in the form of multi-crystalline rather than single-crystal particles. This factor may well have a bearing on the method of preparation of the alloy.

DR. E. W. FELL,† M.Sc. (Member): The authors would, I feel, have added to the interest of the paper if they had given a brief introductory account of the phenomenon of elastic instability in view of its importance in engineering, where members of thin or slender section are subjected to compression and where elastic instability may lead to premature failure of the member by buckling. I shall, therefore, try to explain what is meant by elastic instability and exactly how Young's modulus is of importance concerning it.

The first problems of elastic instability, and which concern lateral buckling of compression members, were solved by Euler about 200 years ago. As an example, we may consider the particular case of a straight and slender

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bar with hinged ends which is compressed by an axially applied load P at both ends (see Fig. E). On increasing the load from zero, the bar remains straight and undergoes only axial or longitudinal compression. For small values of the load, this straight form of elastic equilibrium is stable, i.e. if a lateral force be now applied and a small deflection of the bar produced, this deflection disappears when the lateral force is removed, and the bar becomes straight again. But on further increasing P , a condition is obtained in which the straight form of equilibrium becomes elastically unstable, since a slight lateral force may produce a lateral deflection which does not disappear when the lateral force is removed (see Fig. F). (The condition of elastic instability can be proved by calculation, for the potential energy of the straight form is found to be greater than the potential energy of the laterally deflected form.)



FIG. E.

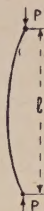


FIG. F.

The axial load P which is sufficient to keep the bar in a slightly bent form (Fig. F) is termed the "critical" load. It is assumed throughout, of course, that the stress in the bar is insufficient to cause any departure from perfectly elastic behaviour of the bar. The "critical" load is given by the equation:

$$P_{\text{crit.}} = \frac{\pi^2 EI}{l^2}$$

where E is Young's modulus and EI is the flexural rigidity of the bar. Thus the "critical" load may be quite low and much less than the load that would cause failure under compression if the bar were to remain straight.

Hence the significance of Young's modulus is that the "critical" load is in simple proportion to it, or, the higher the Young's modulus of elasticity the less is the tendency of the member to fail by buckling.

DR. G. V. RAYNOR: I should like to amplify my remarks made at the meeting by referring briefly to the influence of ternary-compound formation on the Young's modulus of aluminium-rich alloys. If two solutes B and C are separately effective in raising the Young's modulus of the aluminium-rich alloys, then, in the absence of ternary-compound formation, or the formation of compounds between B and C , it would be expected that the effects of B and C present together would be roughly additive. If, however, a ternary compound is formed, marked deviations from additivity may occur.

The system aluminium-manganese-chromium may be taken as an example. Above 590°C ., equilibrium in the solid alloys involves the primary aluminium-rich solid solution, MnAl_6 , and CrAl_7 . On annealing below 590°C ., a ternary compound, denoted G , appears. It enters into equilibrium with the primary solid solution when the manganese content is roughly four times the chromium content; it appears to contain much more aluminium than either MnAl_6 or CrAl_7 , and to have a composition in the region of $(\text{CrMn})\text{Al}_{13}$. Since the value of Young's modulus for this hard compound would not be expected to be very widely different from those of the binary compounds, the slope of the curve of Young's modulus against composition for a ternary section in which the $\text{Mn}:\text{Cr}$ ratio was maintained at $4:1$ would be expected to be markedly steeper than the slope for composition ranges involving only MnAl_6 or CrAl_7 . It was for this reason that the aluminium-manganese-chromium system was suggested as worthy of examination, and it is particularly gratifying to learn from Mr. Dudzinski's subsequent work

that alloys containing manganese and chromium in the ratio 4 : 1 and annealed for long periods below 590°C . do indeed develop abnormally high Young's modulus values, as predicted. The increase in Young's modulus is of the order of 0.52 ton/in.² per unit increase in solute concentration. Alloys in which the Mn : Cr ratio varies from 3 : 1 to 1 : 4 do not, I understand, develop such high Young's modulus values, which is in agreement with the foregoing ideas.

Similar considerations, though on a much smaller scale, might apply to aluminium-iron-nickel alloys, in which, over a certain range of compositions, the primary solid solution is in equilibrium with a ternary compound containing 9 aluminium atoms to 2 solute atoms, as compared with the ratio 3 : 1 for both FeAl_3 and NiAl_3 . In general, if a ternary compound $\text{Al}_x\text{B}_y\text{C}_z$ has a Young's modulus value comparable with those of the binary compounds Al_pB_q and Al_nC_m , it should have a larger proportion of aluminium in it than either of the binary compounds if any improvement over the additive effects of the two solutes is to be obtained.

In some cases an intermetallic compound formed between the two solutes may enter into equilibrium with the primary solid solution over certain composition ranges. Since it would contain no aluminium at all, the effect of such a compound per unit increase in composition would not be expected to be significant, unless it were to possess an abnormally high Young's modulus.

Mr. Dudzinski, in his reply to the verbal discussion, referred to the effects of several other elements, including iron, chromium, and copper. Iron appears to be more effective than would be expected from the heat of formation of FeAl_3 as compared with those of NiAl_3 and Co_2Al_9 ; this may possibly be connected with the reported decomposition of FeAl_3 into Fe_2Al_7 and Fe_2Al_5 on cooling. The effect of copper is small, as would be expected from the lower heat of formation of CuAl_2 , which contains a relatively low proportion of aluminium, while Mr. Dudzinski's figures confirm that chromium is somewhat more effective than manganese, in agreement with my suggestion at the meeting. Tungsten, although it forms an aluminium-rich compound WAl_{12} , appears to be somewhat less effective per unit increase in composition (by weight) than silicon. This is not unexpected, since the atomic weight of tungsten is high; the weight percentage of aluminium in the compound is therefore low, and the effect in raising Young's modulus for the aluminium-rich alloys per wt.-% of the solute is correspondingly small. No thermal data exist for the compounds MoAl_4 , VAl_3 , and TiAl_3 , but all have high melting points, and it is not surprising that molybdenum, vanadium, and titanium are all reasonably effective in raising the Young's modulus of aluminium.

It should be mentioned that conclusions based on the heats of formation and the compositions of intermetallic compounds may be modified by the existence of non-equilibrium conditions, which may lead to the presence, in the experimental material, of compounds containing a smaller proportion of aluminium than the compound which comes into equilibrium with the primary solid solution. The composition factor would tend to decrease the improvement of Young's modulus per unit increase in composition; on the other hand, the heats of formation, and hence presumably the Young's modulus values, are frequently higher for such compounds than for the most aluminium-rich compounds, which would tend to augment the effectiveness of the solute. It is therefore difficult to predict the modifying effect of non-equilibrium conditions, except in special cases.

The AUTHORS (*in further, written, reply to the discussion and in reply to correspondence*): We wish to endorse the plea by Professor Aitchison for a

greater knowledge of the properties of constituents. It is our opinion that this will be a fruitful approach to a prediction of the physical, chemical, and mechanical properties of new alloys, and work was once started at the Royal Aircraft Establishment to produce compound crystals of a suitable size and shape. It is considered that a study of the effect of constituents on the stress distribution in a matrix would be a step in this direction.

With regard to the suggestion by Mr. Rotherham that extrapolation of the curves to 100% intermetallic compound gives an approximate value of E for the compound, it is pointed out that no reliance can be placed on such figures, as the linear relationship only holds good for small percentages of the second constituent.

As suggested by Mr. McLean, the slight variation in the values of E for aluminium by extrapolation to zero concentration may be partly due to the effect of solid solution, but there may also be small differences due to differences in soundness of the cast alloys.

In conclusion, we would again emphasize that in our preliminary work, no attempt was made to develop alloys with high E combined with good working properties and high tensile properties. The estimated properties of alloys which, it was suggested, could be obtained were very conservative, but it is our conviction that these should be considerably improved by a systematic development process. We have only to remember the amazing development in the Duralumin type of alloy which has taken place in the last 35 years. Would Wilm have dared to suggest in 1912 that aluminium alloys would be produced with a 0.1% proof stress greater than 40 tons/in.² and a tensile strength greater than 45 tons/in.²?

